

Practical Aspects of the Identification of Cassiterite (SnO₂) by the "Tinning Test"

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*But somehow he hasn't got hold of it quite
Or the liquid you pour on it first isn't right
So that's why he works at it night after night
'Til he knows he can do it for certain.*

(A.A. Milne—"The Alchemist")

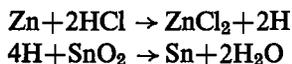
Abstract: It is pointed out that no completely adequate published accounts of the so-called "tinning test", which is used as an aid to the identification of cassiterite, are known to the writer. All of them lack important details and many contain statements that are wrong.

What is believed to be a correct comprehensive account of the test under review is given, and certain other *related* qualitative and semi-quantitative tests for tin occurring as cassiterite are described.

VARIATIONS OF THE "TINNING" TEST

The so-called "tinning test" is one frequently carried out in the field, mill, and laboratory as a rapid and simple aid to the identification of cassiterite, generally by those concerned with the examination of samples of loose grains. The same aid can also be used to identify cassiterite in hand specimens and thin- and polished-sections.

The test may be carried out in a number of different ways, but it may be effected, and often is, by placing the sample in a zinc tray and then covering it with dilute (c. 5N) HCl. After a few minutes any "clean" cassiterite present is usually covered with a grey *matt* coating which on rubbing with, say, a piece of cloth, assumes a bright silvery appearance. This coating has been generally thought to be metallic tin produced by the reduction of the SnO₂ by nascent hydrogen in accordance with the following equations:—



However, the reaction is not as simple as this, as "nascent" hydrogen, generated by the reaction between a dilute acid (say HCl) and certain metals other than zinc will not result in the cassiterite being tinned. Data presented elsewhere in this paper serve to confirm this statement.

The fact that at least one author (Dana, 1932, p. 497), and contrary to general belief, thought that the coating was zinc, was recently brought to the writer's attention, and so he decided to carry out a little library research in order to check what others had to say about this test which is in such common use throughout the tin-fields of the world. The results were most interesting in that none of the writers consulted had described the test and its limitations accurately and completely! Some

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descriptions, such as those of Beringer (1931, p. 220), Betekhtin (p. 289), and Jones and Fleming (1965, p. 21) are correct as far as they go, whilst all the others are to some extent incorrect! Holmes (1930, p. 293) for example, states that the cassiterite should be *heated* with HCl and zinc: this is wrong; cold dilute HCl should be used.

Several writers [Griffiths (1960, pp. 4–5); Davidson (1937, p. 13); Ng and Yong (1969, p. 1122)] say that the cassiterite grains, when treated with zinc and dilute HCl, become silvery when, in fact, the coating is a grey, matt one. Harrison (1954, p. 44) and Griffiths (1960, pp. 4–5) report that the test is an 'infallible' one for cassiterite: others [Palache *et al.* (1944 p. 577); Dana (1932, p. 497); Holmes (1930, p. 293); Davidson (1937, p. 13); Muller *et al.* (1969, p. 563); Ng and Yong (1969, p. 1122)] whilst not stating that all cassiterite reacts positively to the test, imply that this is so. The test is not an infallible one even for 'clean' cassiterite, and Betekhtin (p. 289) is correct when he says that it is "... almost always successful", and so is Beringer (1931, p. 220) who mentions that the test is quite a useful one "... but does not unfortunately act in every case".

It is reasonable to assume that both Betekhtin and Beringer were considering the behaviour of 'clean' cassiterite. That coated cassiterite would not respond positively to the test unless the coat, depending on its nature, were removed before, or during the test, would seem to be a glimpse of the obvious. Nevertheless, the writer once saw considerable consternation generated on a mining property in this country when the test failed because the thin veneer of iron oxides, which was coating the grains, had not been first removed by boiling the sample with concentrated HCl as Jones and Fleming (1965, p. 21) but no other writers consulted, suggest.

It is pertinent to remark here that coatings other than iron oxides may slow down or even prevent the tinning of cassiterite unless preliminary steps are taken to remove them. Amongst such coatings may be mentioned flotation reagents, palm oil (added, on occasion, by Nigerian tributaries to their cassiterite/columbite concentrates to improve their appearance) and thin transparent mineral veneers, such as one found by the writer on free faces of cassiterite from several Malaysian localities and from Beralto (Portugal), which fluoresces a dull orange under short-wave ultraviolet light, and which may be apatite.

Because of the possible presence of undesirable coatings, and because, in addition, sulphide-rich grain samples are prone to react in a somewhat indifferent manner to the test under review, it is advisable, on many occasions, to first subject the sample to the following acid clean. Place the sample (say, 2–3 g) in a small beaker and digest slowly with c. 10 ml of a mixture of HNO₃ and HCl (2:1 for sulphide-rich samples and 1:3 for 'oxide' ones). After the brown fumes have ceased to be evolved, add 5 ml HCl, cover with a watch glass, and boil for 5 minutes. Cool, dilute, wash by decantation, and then carry out the tinning test.

ZnO coating on the zinc tray (or on the zinc block with a depression to hold the sample) will slow down the tinning process and should be removed with, say, a wire brush, before carrying out the test.

Most writers who have discussed the tinning test make no mention of the fact that it can be used as an aid to the identification of cassiterite in polished section. Muller *et al.* (1969, p. 563) note that the test "may readily be extended to polished ore specimens by applying a paste of fine zinc powder and adding acid". Beyond

doubt this is the only satisfactory way of tinning such specimens if they are embedded in plastic mounts of the usual sizes, or if the specimen under examination is not embedded, but has a polished surface of, say, 0.5 sq. in. or more.

The writer makes a thick paste of zinc powder and water and places a layer of it of 2 to 3 mm thickness over the surface of the specimen. He then adds 5N HCl by means of a teat pipette, at intervals of about 5 seconds, for a period of one minute, and in such a way that hydrogen bubbles are constantly generated over the whole of the covered surface. When the minute is up the remaining zinc is removed with a jet of water. This procedure will result in all 'normal' cassiterite being tinned if the surface under examination is initially clean. It is not enough that the surface looks clean, it really must be chemically clean and, in particular, free from films of oil or grease (that are all too easily acquired). These and other films may be removed from rock slices by rubbing them briefly on an iron or glass lap charged with 600 carborundum and water. Polished sections may be cleaned, in the usual way, with acetone, or, perhaps better, by buffing them, say, on a slowly rotating (c. 100 r.p.m.) sanded Nylon velvet lap charged with freshly ignited magnesia and water.

The tinning test involving the use of zinc paste, and carried out in exactly the way described above, may also, on occasion, be used to establish the identity of cassiterite in thin section, and is particularly useful when there is some doubt as to whether certain small components are, or are not, cassiterite. This technique, which was introduced to the writer by Mr E.H. Davison in the thirties, was, surprisingly, not mentioned by Davidson in the work referred to above.

It is important to observe that thin and polished sections containing appreciable calcite will be severely damaged by the test.

At this stage it seems relevant to note certain other details of the tinning test that are ignored by all the writers mentioned earlier:—

- i. The metallic coating developed on the cassiterite as a result of the test is, indeed, tin. This can be readily demonstrated by removing some of it, after washing the sample for c. 5 minutes in running water, dissolving a little of it by warming it with concentrated HCl on a microscope slide, and then subjecting aliquots to the rubidium chloride test for tin, and the ammonium mercuric thiocyanate test for zinc in the manner described by Short (1940). Only the tin test will give a positive reaction. Further confirmation can be readily obtained by warming a 'tinned' grain on a microscope slide in a drop of concentrated HCl. The drop is then added to a drop of a saturated aqueous solution of cacotheline. The development of a violet coloration proves the presence of stannous tin, and that could only have been derived from the metallic coating. (See Feigl, 1954, pp. 104–105.)

In addition, as the writer's colleague Mr E.B. Yeap has demonstrated, the optical properties of the coating, as noted in reflected light, under the microscope, are those of tin, not zinc.

- ii. Grains of Nigerian, black, magnetic cassiterite, when heated in, say, a tin furnace at c. 1,000°C for 30 minutes become red, and somewhat more transparent, on cooling, and lose their magnetic property (MacLeod and Jones (1955): in these respects it differs from columbite, with which it is often associated, and such treatment has been regarded by some as a possible starting point for the

separation of these two species in the mill. Such treated cassiterite, when subject to the normal tinning test, will *not* tin. However, according to Mr E.K. Furze (personal communication) if this heat-treated cassiterite is boiled with a dilute solution (a few percent) of NaOH for a few minutes, and then washed, its capacity for being tinned is restored.

Generally speaking, heat-treated cassiterites, from other areas, vary in their response to the tinning test. Several Cornish samples, for example, which were tested by the writer, did tin, but at a very reduced rate. This is a feature, also, of some of the Cornish cassiterite which has been derived from calcined mill-products.

That certain cassiterites, which have not been modified by man, may not react positively to the tinning test, is, doubtless, in part due to the fact that they have been subject to natural heat treatment, occasioned by forest fires, or volcanic action. The ruby-coloured cassiterite found in the Nigerian placers is due in all likelihood, to one or other of the above causes, whilst the orange-vermilion cassiterite, occurring locally in Mexico, is due, probably, to the latter. These highly coloured cassiterites are not always readily tinned.

iii. A number of variations of the tinning test, as described above, that have been investigated, need to be mentioned. Some of these are effective whilst others are not.

(a) Instead of a zinc tray, the test may be carried out in a saucer, a beaker, a test-tube, or even a dulang, provided zinc powder, or mossy zinc (although powder is to be preferred) is used. When using zinc powder to examine grains, it is best, in order to avoid misidentification, to add enough acid to dissolve all the zinc.

Betekhtin (undated p. 289) suggests the following variation of the tinning test that, in this writer's view, might be a particularly convenient one to apply, on occasion, in the field:—"If a drop of hydrochloric acid is placed on cassiterite and touched with a piece of zinc (or better with a specially made zinc needle), after a while, a metallic coating will appear on the cassiterite."

(b) An aluminium tray, scrubbed with a wire brush, can be substituted for the zinc one, but not more than 2-3N HCl should be used otherwise the reaction becomes too violent and, perhaps for mechanical reasons, the cassiterite will not tin.

(c) Boiling a cassiterite concentrate in a test-tube with 1:1 HCl and iron filings (which were somewhat impure as some H₂S was generated initially) for 5 minutes did not result in the cassiterite being tinned.

A similar test was carried out in a small beaker, excepting that the iron consisted of 24 pieces of wire (diameter c. 1.5 mm) each piece 1.25 cm in length. When 1:1 HCl was used, none of the cassiterite grains were tinned after a treatment lasting 5 minutes and when 1:1 H₂SO₄ was substituted for the HCl, again, no tinning occurred. Attempts to tin cassiterite in an iron tray failed when either near-boiling 1:1 HCl or 1:1 H₂SO₄ were used. Then four samples of cassiterite were placed, together with iron filings (as supplied by B.D.H., Ltd.) on separate watch glasses. 5N HCl was added to the first. 1:1 HCl to the second, 5N H₂SO₄ to the third, and 1:1 H₂SO₄ to the fourth. Hydrogen, plus some noxious gases, were evolved without any heating, at

a moderate to slow rate, and a rather stable froth built up in each case. After 10 minutes, and when the acids had been decanted, the samples were examined. Not a single fragment of cassiterite had tinned.

Finally, coarse grains of clean cassiterite were placed in an iron crucible and then covered with 5N HCl. The crucible was heated intermittently, to ensure a constant and rapid generation of hydrogen, for ten minutes. At the end of this period the cassiterite completely lacked a coating of tin. This same cassiterite, when placed in an old zinc tray, and covered with 5N HCl, was tinned in 15 seconds!

These tests also prove quite conclusively that the following note concerning the tinning test, which appears in the Encyclopaedia Britannica (22, p. 236, 1962) is in part incorrect. It states, "a chemical test that distinguishes it (cassiterite) from those minerals with which it may be confused is the treatment of the mineral with *cold dilute* hydrochloric acid or sulphuric acid in the presence of *zinc or iron*. This reduces the surface oxide to a gray coating of metallic tin which can be washed and rubbed to a silvery surface". It is at least satisfactory to read the last sentence: that certainly is correct!

Jones (1925, pp. 36-37) having given a reasonably adequate description of how natural cassiterite may be identified by tinning it by the employment of zinc and either dilute HCl or HS_2O_4 , goes on to say that "if zinc is not available, iron filings or nails can be used, but the acid in that case must be heated until bubbles of hydrogen are evolved." Again, the writer's tests, that are noted above, indicate that iron cannot be used instead of zinc, even when the acid is heated.

Jones concludes his remarks about the tinning test by stating that "..... no other mineral treated in this manner will give this bright silver-white coating". This may well be correct, but the writer wonders if the comparatively recently discovered mineral starrigite, many of whose properties are so similar to those of cassiterite that originally starrigite was identified as cassiterite, would behave positively to the tinning test. Unfortunately he has no starrigite so he has not been able to test this possibility.

- (d) A sample of cassiterite treated in a test-tube with 5N HCl and pieces of magnesium ribbon did not tin. This was possibly because the magnesium floated and so the liberated hydrogen did not come into contact with the SnO_2 . However, even when a small pebble of cassiterite was subject to this test, having been first placed in a 'basket' of magnesium ribbon, it also did not tin.

Possibly if tests (c) and (d) were further modified they *might* be used instead of the zinc one. However, these tests, as carried out, indicate that however they may be modified, the great superiority of the zinc ones over all the others will be maintained.

- (e) The zinc test may be modified to accelerate the rate of tinning by first adding a drop or so of lead acetate solution to the zinc tray and then washing the latter before starting the test. That a lead/zinc couple is more effective than zinc alone can be readily demonstrated by covering the floor of the zinc tray with cassiterite grains then covering the middle third with a piece of lead foil and adding 5N HCl. When the uncovered cassiterite grains are just beginning to tin those under the lead will be completely tinned.

- (f) Dilute H_2SO_4 may be substituted for dilute HCl when carrying out the tinning test, and if the test is carried out in a beaker or test-tube, a saturated, boiling aqueous solution of potassium bisulphate may take the place of these acids.
- (g) Cassiterite may also be tinned by electrolytic means. One way of doing this is to make a depression on a block of graphite, place the sample therein and cover it with water acidified with H_2SO_4 . Pass a modest current for 5 or 10 minutes through the system, making the graphite block the cathode, and using a carbon rod, dipping into the electrolyte, as the anode.

FURTHER USES OF THE TINNING OF CASSITERITE

- (i) Years ago the Russians devised a test (Sokoloff and Hawkes, 1950, pp. 65–66) for the estimation of tin, as cassiterite, in stream sediment samples during geochemical prospecting that initially involved tinning any cassiterite present in the sample. The instructions given by the Russians for carrying out the test, together with certain data re the results obtained by them are as follows:—

“One cc of ground sample is treated for 30 minutes with 3 to 4 cc of strong hydrochloric acid; 0.5 g chemically pure zinc is then placed in the crucible. After 2 or 3 hours the liquid is tested for tin. For this purpose, the lower end of a test tube filled with water is immersed in the crucible. After the contents of the crucible are mixed, the wet lower end of the test tube is introduced into the reducing cone of the flame of a Bartle burner. A typically blue-colored flame envelopes the test tube. The area covered by the flame is proportional to the tin content of the sample. A quantitative estimation of tin is made by comparison with standard solutions. Table 14 shows comparisons between results of the above method and of the chemical analysis of a “coded” series of samples from one of the localities”.

(Table 14 of Sokoloff and Hawkes)

Comparison between flame tests for tin & chemical analyses

Method	Percent tin												
	0.01	0.01	0.1	0.1	0.1	0.1	0.1 to 0.5	0.1 to 1.0	More than 0.1	More than 1.0	1.0 to 3.0	1.0 to 3.0	0.1 to 0.5
Flame tests													
Chemical	0.02	0.04	0.07	0.08	0.1	0.16	0.22	0.33	0.36	1.28	2.47	3.74	0.44

“Mass examination permits a high productivity for the testing part of the work.”
For further data concerning the flame test for tin see Feigl (1954, p. 105).

- (ii) In 1969 Hak *et al.* gave the following, somewhat vague details of a new method of quantitative mineralogical determination of cassiterite in alluvial concentrates, in which tinning the stannic oxide played an essential role. “A weighed sample of the rough concentrate in which the grains of cassiterite, through reaction of SnO_2 with hydrogen, were coated with a film of metallic tin was subjected to low-intensity magnetic separation. The magnetic field intensity must be sufficiently low to prevent the separation of tin in the magnetic fraction. The non-magnetic fraction was then subjected to galvanising by ferromagnetic material. The step could be so performed that the ferromagnetic film was deposited

only on those cassiterite grains that had a film of metallic tin. The thickness of the ferromagnetic film, too, must be controlled so as to allow all cassiterite to pass over into the magnetic fraction during the second magnetic separation. The magnetic field intensity, used in the second separation, was lower than during the first one. The cassiterite contents in the rough concentrates could be computed after the fractions deprived of cassiterite were weighed". The writers go on to say that employment of this method has greatly accelerated their rate of evaluation of the cassiterite content of samples.

Clearly, such a method would be most useful to all those concerned with the determination of the cassiterite content of samples of grains, and it is a great pity that Hak and his co-workers were not prepared to give full details of the process. The writer, years ago, attempted to place a coating of iron on tin-coated cassiterite, in order to facilitate separation of the latter from grains of quartz, etc., but without success. The method of iron deposition tried was that of Vincent (1951) who had successfully separated conducting from nonconducting mineral grains by placing them in an iron cell partly filled with an aqueous solution of calcium chloride and ferrous chloride and passing a current of 0.05A per sq. cm. through the system for 3 or 4 minutes. This resulted in the conducting grains being coated with iron, and this provided a ready means of separating them from the non-conducting fraction.

The statement of Hak and his co-workers revived the writer's interest in this problem of separation, and by good fortune he mentioned the subject to Mr J.H. Harris, the Project Manager of the Ore Dressing Research Division, U.N.D.P., Manggar, Belitung. Mr Harris was able to give details of Hak's method and to comment on its value, as the possibility of using the method in Indonesia had been investigated in 1971. So the following details can now be added to the description of the method that is given above:—After the cassiterite is tinned and the magnetic fraction has been removed, the non-magnetic fraction (containing the tinned cassiterite) is subject to a galvanic plating processes by means of which the tinned grains are coated with nickel. This process takes from 15 to 20 minutes, and when it is completed the sample is washed and dried and the coated grains are separated by a magnet. Essentially the nickel plating involves the use of a copper dish cathode. (the sample is placed in this), a nickel plate anode, a nickel-containing electrolyte and a stirrer (to ensure that all the grains come into contact with the cathode). Apparently Hak found the method worked very well when 'coarse' (average 35-mesh) Nigerian samples were subject to it but it failed to yield satisfactory results when minus-48-mesh Bangka material was the test substance.

The following comments of Mr Harris on Hak's method serve to highlight the inherent defects of this and similar methods of mineral separation:—

- “(a) When tinning cassiterite by the use of zinc powder it is *not* easy to coat *all* cassiterite particles.
- (b) When dissolving the excess zinc in acid it is not easy to be sure when the solution is complete. If any zinc is left it will later be nickel-plated and be collected with the cassiterite. If boiling is prolonged to ensure solution of the zinc, then some of the tin coating on the cassiterite will be dissolved off and those grains will *not* be electroplated and therefore *not* recovered. If the cassiterite grains are very small they may be totally reduced to tin and subsequently completely dissolved during the boiling and therefore lost.

- (c) In the magnetic separation prior to nickel-plating, magnetic cassiterite would be lost. This may be 10 per cent or more of the total. (Mr Harris is here referring to Indonesian samples.)
 - (d) In the nickel-plating, *all* conducting minerals are liable to be coated, particularly sulphides and rutile, and will therefore count as cassiterite in the second magnetic separation.
 - (e) The floor of the cathode also becomes plated and the throwing power of the electrolyte is sufficient rapidly to cover over small conducting particles and weld them to the floor. In Hak's apparatus the nickel probably deposits at 10 microns per minute or more. In three minutes 30 microns would deposit and I imagine this would be sufficient to trap and hold a particle of 100 mesh (150 microns) in spite of the stirring. I have not thought of a way to release these stuck bits.
 - (f) The second magnetic separation suffers from the same disadvantages as any other, in that it must be less than 100 percent efficient, especially with fine grains".
- (iii) Minor amounts of cassiterite associated with ilmenite, etc., in a sample of grains, can be conveniently determined by subjecting a weighed amount of the sample to the tinning test in a zinc tray. After the completion of the tinning and the addition of water, the then somewhat buoyant cassiterite is persuaded, by agitation, to migrate to one corner—an operation which is usually not difficult to carry out. The corner of the tray is bent down and the cassiterite is washed off. The cassiterite-free fraction is then removed, washed, dried, and weighed, and the cassiterite present is determined by difference (Hosking, 1956). (Footnote.)
- (iv) Ramdohr (1969, p. 991) notes that "good structural etching (of cassiterite), which may also serve as a diagnostic feature against rutile, wolframite, tantalite, columbite, uraninite, etc., is obtained with nascent hydrogen (accord. to Piepmayer). The polished section is immersed in a bowl containing a piece of zinc metal and some tin granules in diluted HCl. A thin film of tin is precipitated on the cassiterite, under which good etching is visible after dissolving the tin with HNO₃. Reaction needs only little time (5 min.); longer with higher Fe-contents".

By employing the method as described, successful tinning would only be achieved if the specimen were very small and its polished surface, say 1/16th sq. inch in area, rested on the zinc. Adequate tinning would not be effected even in this case if the surface area of the polished section were comparatively large, say ½ to 1 sq. inch, regardless of whether one was concerned with a single unmounted specimen or a briquette. When a large specimen is placed with its polished surface in contact with the zinc base of a zinc tray and aliquots of 5N HCl are added to ensure constant active evolution of hydrogen for 5 minutes some of the cassiterite at the edge of the polished section *may* be tinned but commonly none of the cassiterite present is so affected.

To obtain good structural etching of cassiterite use the zinc paste method described earlier (and the usual reduction period of one minute) then dissolve the tin in 1:1 HNO₃ (the metal disappears almost instantly).

Footnote: The referee of this paper noted in his comments that he had endeavoured, but without success, to recover tinned cassiterite grains in a composite sample by entrainment of the former by means of mercury.

- (v) Recently Mr. M. Jones (of B.H.P.) brought to the writer's notice the fact that a few years ago Lever (1965) designed a test for the 'detection of small amounts of cassiterite in pan concentrates' which was an elaboration of the tinning test. Briefly, the test is carried out as follows:—

A small sample, c. 0.05g. of the test material, is subject to the usual tinning procedure and then it is washed well and dried. The treated sample is next dispersed on a piece of dry, starch-impregnated, chromatography paper and the whole is moistened, not flooded, with 1:1 HCl. The grains are brushed off the paper when the latter is sufficiently dry and then the paper is sprayed with a 2 g.p.l. solution of iodine.

This treatment causes the paper to become blue excepting where grains of tinned cassiterite have rested! These later spots remain white, at least for an hour or so.

Lever claims that this permits the detection of a single grain of cassiterite in, say, what is essentially an ilmenite concentrate, and even if the grain in question is very small. He also notes that, in addition, ilmenite, magnetite, tourmaline, pyrite, arsenopyrite, chalcopyrite, stibnite, bismuth, copper, and mild steel chips, do not interfere.

Finally, the writer has shown that chromographic contact prints of tinned cassiterite in rock slices and polished sections may be made by making use of the reactions employed by Lever. This possibility was also suggested to the writer by Mr. Jones.

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