Bead Tests

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Synopsis

IN this review — which is written to fill an obvious gap in the literature — the following tests are described and their merits appraised :—Classical bead tests : Feigl's bead test for gold : tests depending on the examination of beads under ultraviolet light.

Bead tests are of unquestionable value to the mineralogist, yet no text book, nor paper — so far as the writer is aware — deals comprehensively with them. This fact justifies this review.

Classical bead tests.

Classical bead tests depend on the fact that when small quantities of certain elements or their compounds are fused with an appropriate flux — borax, microcosmic salt or soda — under oxidising and/or reducing conditions, beads are obtained, either of characteristic colours, or containing characteristic inclusions.

In practice, a bead of flux is built up in a platinum wire loop or on a magnesia rod and is dipped, whilst still hot, into a very small quantity of the powdered test-substance. The mixture is then fused in either the flame of a bunsen burner or in the blowpiped flame of an alcohol, or similar lamp.

Whether the fusions are conducted in the oxidising or in the reducing zone of the flame, the colour of the bead is noted when it is hot and after it has cooled, as these colours often differ, and both are of diagnostic value.

The chemical bases of these tests are as follows (see Vogel, A. I., 1947, 112-114) :—

1. The oxides of several elements form coloured metaborates and orthophosphates when they — or salts from which they can be derived — are fused under oxidising conditions with borax and microcosmic salt respectively.

2. In the reducing flame metaborates and orthophosphates of the elements under test are probably formed initially, but may subsequently be reduced to other compounds or to elements, which impart both to the hot and cold beads colours which often differ from those appearing under oxidising conditions.

3. Silica, which is liberated when certain silicates are strongly heated with microcosmic salt in the oxidising zone of the flame, does not dissolve in the melt and appears in it as a "skeleton". This aids the detection of silicates. A typical reaction is of the type

$$CaSiO_3 + NaPO_3 = NaCaPO_4 + SiO_2.$$

As certain silicates dissolve completely in fused microcosmic salt, the absence of a skeleton is *not* definite proof of the absence of silicate in the test substance.

4. When a bead of sodium carbonate is, after moistening, dipped first into a little powdered potassium nitrate and then into a small quantity of powdered manganese compound and the whole is heated in the oxidising flame, a green bead of sodium manganate is formed :—

$$MnO + Na_2CO_3 + O_2 = Na_2MnO_4 + CO_2.$$

Chromium compounds similarly treated result in the production of yellow sodium chromate :—

$$2Cr_2O_3 + 2Na_2CO_3 + 3O_2 = 4Na_2CrO_4 + 4CO_2.$$

The general course of the reactions involved in the production of characteristically coloured borax and microcosmic salt beads may be appreciated by considering the reactions — briefly outlined below — between a copper salt and the above fluxes under various conditions.

1. **Borax Bead.**

(i) **Oxidising conditions.**

(a) $Na_2B_4O_7.10H_2O \xrightarrow{\text{Heat}} \underbrace{2NaBO_2 + B_2O_3 + 10H_2O.}_{\text{Colourless.}}$

Sodium Metaborate. Boric Anhydride.

(b) Copper salt $\xrightarrow{\text{Heat}} CuO.$

(c) $CuO + NaBO_2 \longrightarrow NaCuBO_3.$ (orthoborate)

The product is pale green

(A probable reaction.)

(d) $CuO + B_2O_3 \longrightarrow Cu(BO_2)_2$

(e) $Cu(BO_2)_2 + 2NaBO_2 \longrightarrow Na_2[Cu(BO_2)_4].$

! (A possible reaction.)

hot and blue-green cold.

(ii) **Reducing conditions.**

(a) $2Cu(BO_2)_2 + 4NaBO_2 + C \longrightarrow Cu_2(BO_2)_2 + Na_2B_4O_7 + CO.$

(Colourless)

The product is pale-green hot and red and opaque cold.

Note :—The carbon is derived from the flame.

(b) $2Cu(BO_2)_2 + 4NaBO_2 + 2C \longrightarrow 2Cu + 2Na_2B_4O_7 + 4CO.$

2. **Microcosmic Salt Bead.**
Oxidising conditions.

(a) $Na(NH_4)HPO_4$ (Microcosmic Salt.) —Heat→ $NaPO_3 + H_2O + NH_3$ (Metaphosphate. (Colourless.)) — The product is dark-green hot and blue cold.

(b) $NaPO_3 + CuO$ ——> $NaCuPO_4$. Orthophosphate. (Coloured.)

Bead tests of the type under review are of great value to the mineralogist because they are both simple and rapid and only a very limited amount of apparatus and a small number of reagents are needed. Furthermore, a considerable number of economically important elements can be readily detected by their use, as a glance at Table 1 will indicate. They are normally used in conjunction with charcoal block and other " blowpipe tests ". A difficulty arises when testing a sample containing two or more elements which are capable of producing coloured beads with the fluxes noted above. Either the colour of the bead will be so modified that it is not characteristic of any of the elements present, or the colour due to one element will completely mask those due to the others. Thus, a borax bead prepared from a sample containing 5 or 6 times as much nickel as cobalt will, nevertheless, be blue. Table 2 indicates some of the bead colours encountered when more than one " colour-producing " element is present and clearly demonstrates the short-comings of the method under such circumstances.

On the other hand, the addition of substances to a given bead sometimes assists identification. Thus, the presence of manganese in a microcosmic salt bead is confirmed if on dipping the *hot* bead into powdered potassium nitrate a purple, swollen mass develops. When a tungsten compound is fused with microcosmic salt in the reducing zone a blue glass — or bluish-red one if iron is present — develops. The intensity of this colour may be considerably increased by re-heating the bead together with a small fragment of tin foil in the reducing zone.

Cation	Minimum amount detectable (gammas)	
	Borax	Microcosmic Salt
Chromium — Oxid. flame (O.F.)	1.8	1.8
Manganese — O.F.	18.8	—
Nickel — O.F.	5.0	7.5
Cobalt — O.F.	0.5	1.0
Copper — O.F.	24.8	24.8
Copper + $SnCl_2$ — Red. flame	1.9	9.9

TABLE 1.

A Summary of the Classical Bead Tests.

(After Davison, E. H. Field tests for minerals. Chapman and Hall, London. 1937, 10.)

BORAX BEADS

Metal	Oxidising Flame		Reducing Flame	
	Hot	Cold	Hot	Cold
Copper	Pale green	Blue-green	Pale green	Red
Cobalt	Blue	Blue	Blue	Blue
Chromium	Orange	Yellow-green	Green	Green
Cerium	Yellow	Pale yellow	—	—
Iron	Yellow	Colourless	Pale green	Colourless
Manganese	Violet	Violet	Colourless	Colourless
Nickel	Violet	Red-brown	Grey	Grey
Titanium	Pale yellow	Colourless	Grey	Violet
Vanadium	Yellow	Pale yellow	Green	Green
Uranium	Orange	Yellow	Green	Pale green

MICROCOSMIC SALT

Metal	Oxidising Flame		Reducing Flame	
	Hot	Cold	Hot	Cold
Copper	Dark green	Blue	Green	Red
Cobalt	Blue	Blue	Blue	Blue
Chromium	Green	Green	Green	Green
Cerium	Yellow	Colourless	Colourless	Colourless
Iron	Yellow to red	Yellow to colourless	Yellow-green	Colourless
Manganese	Violet	Violet	Colourless	Colourless
Molybdenum	Yellow-green	Colourless	Green	Green
Nickel	Brown-red	Reddish-yellow	Brown-red	Reddish-yellow
Titanium	Yellow	Colourless	Yellow	Violet
Tungsten	Yellow	Colourless	Blue	Blue
Vanadium	Yellow	Pale yellow	Green	Green
Uranium	Yellow	Yellow-green	Green	Green

TABLE 2.

COLOURS WHICH BORAX BEADS MAY ASSUME WHEN MORE THAN ONE "COLOUR-PRODUCING" ELEMENT IS PRESENT

Elements	Oxidising Flame		Reducing Flame	
	Hot	Cold	Hot	Cold
Mn and Fe	Violet to blood-red	Brownish-violet	Yellow	Bottle-green
Mn, Fe and Co	Plum colour	Plum colour	Bluish-green	Blue
Mn, Fe, Co and Ni	Green	Greyish-blue	Blue-green	Green
Fe, Co and a little Ni	Yellowish-green	Green	Greenish-blue	Blue
Co and much Ni	Violet-brown	Brown	Blue	Blue
Fe and Co Fe and Cu Fe and Ni	Green	Light green, blue or yellow according to relative quantities		

Augusti and Pascalino (1937, 166) report the following sensitivities of borax and microcosmic salt bead tests and conclude that only cobalt, chromium and copper (in the presence of $SnCl_2$) can be detected in the small amounts which interest the micro-chemist.

Feigl's bead test for gold.

In order to detect gold in solution Feigl (1947, 101) evaporates a little of the liquid in a capillary tube and then by fusion converts the tube into a glass bead. Depending on the quantity of gold present, either a yellow metallic, or a purple-red filament is seen in the centre of the bead which acts as a lens. Sometimes the writer (Hosking. Unpublished studies.) has found that the whole bead is coloured a faint purple-red.

Tests depending on the examination of beads under ultraviolet light.

Groves (1951, 250-251) describes in considerable detail a very sensitive fluorescent bead test for the identification of uranium in certain minerals (e.g., uraninite and carnotite) which do not fluoresce under ultraviolet light. Pure sodium fluoride is fused in a platinum loop and then a little of the finely powdered test substance is introduced and strongly heated in the bead for 2 or 3 minutes.

The cold bead is then examined under ultraviolet light — preferably of long wave-length — when the presence of uranium is indicated by a bright lemon-yellow or yellow-green fluorescence. Rare-earths, not infrequently present in refractory uranium species, tend to quench the fluorescence, and " . . . lime interferes very seriously (less so Si, Ti, Fe and SO_4) ; as little as 6 per cent. of calcium fluoride in the bead completely extinguishes the fluorescence ". (Groves, op. cit., 251.) Niobium-bearing minerals which do not contain uranium give, when treated in the above way, a bead which fluoresces a pale, dull-yellow. Groves' uranium test suffers from the further disadvantage in that sodium fluoride, which is generally regarded as the most suitable flux for preparing a uranium bead which will fluoresce when excited by ultraviolet light, has a high melting-point (980° C.) which is difficult to obtain under field conditions.

TABLE 3.

A SUMMARY OF SMITH'S FLUORESCENT BEAD TESTS.

(After Smith, O. C., 1953, 60.)

1. **Borax beads examined under short-wave ultraviolet light.**

Uranium ...	Oxidising flame	Greenish.
Copper	Reducing flame	Pinkish.

2. **Microcosmic salt beads examined under short-wave ultraviolet light.**

Uranium ...	Reducing flame	Greenish.
Copper	„ „	Reddish.
Tungsten ...	„ „	Pinkish.

Note :—No other borax or microcosmic salt beads respond.

3. **Sodium fluoride and lithium fluoride beads prepared in the oxidising flame and examined under both long-and short-wave ultraviolet light.**

(i) **Sodium fluoride beads.**

	Short wave.	Long wave.
Bismuth	... Blue-white	Yellow.
Columbium	. Blue-white	None.
Titanium	... Light green	None.
Tungsten	... Light bluish-yellow	None.
Uranium	... Brilliant lemon-yellow ...	Bright yellow.

(ii) **Lithium fluoride beads.**

Bismuth	... Orange	Dark orange.
Columbium	. None	None.
Titanium	... Dark green	None.
Tungsten	... Light blue	None.
Uranium	... Brilliant blue	Blue-green.

Smith (1953, 60) has extended the fluorescent bead test technique to the identification of several other elements and his tests are summarised in Table 3. The writer has found (Hosking. Unpublished studies.) that most of these are only reliable for identifying elements in fairly pure, simple substances, and is of the opinion that considerably more work is necessary in order to evaluate the degree of interference due to the presence of varying amounts of other elements before the true value of these tests to the mineralogist can be assessed.

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