







FIRE ASSAY SL – Carrer de Novell, $54-2^{nd}$ Floor OFFICE S-08014 BARCELONA - SPAIN - EU

WARE-HOUSE ES-08389 PALAFOLLS – BCN – SPAIN – EU

FIRE ASSAY METHOD: BASIC INFORMATION

Edition October 2024



WOODCUTS

& TEXTS















Juan de Arfe

Lazarus Ercker

Georg Bauer "Agricola"

Bauer

<u>Arfe</u>

Ercker

5.3

5.4

5.5

(Basel, 1556)



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BIBLIOGRAPHY: TEXTBOOKS INTERNET ARCHIVE (I A) (free download!) GOLD ASSAY BEAUTIES: POEMS &	INDISPENSABLE BOOKS	• 4. REFERENCE BOOKS - Free download	Bugbee Shepard Fulton Smith Rose1 Rose2 Bering. Gilbert Anders. StJordi	4.1 4.2 4.3 4.4 4.5 4.6 4.7 4.8 4.9	

♦ 5.4 "Quilatador de la Plata, Oro y Piedras", (Valladolid, 1572)

♦ 5.5 "Das große Probierbuch", (Frankfurt / Main, 1598)

♦ 5.3 "De Re Metallica",

AGRICOLA









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ESSENTIAL FIRE ASSAY TERMS

Oct-2024 Terms01 Assay Ton.doc



FLUXING

FUSION

SLAG Separation **CUPELLATION**

PARTING

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MEANINGS & CLUES

CITATIONS FROM AUTHORITATIVE TEXTBOOKS & REFERENCE MANUALS

"ASSAY-TON"

AUTHOR CONTEXT

> In weighing the materials for a crucible charge the use of a set of assay-ton weights saves much labour in calculation. The assay-ton is a weight which contains as many milligrammes as a ton contains ounces.

Rose1

Thus an English or long ton of 2,240 lb ... contains 32,666 Troy oz, so that the corresponding assay-ton must weigh ... 32.666 g. If the weight of the resulting bead of gold (or silver) from an assay-ton of ore is 1.5 mg, then the ore contains 1.5 oz of gold per statute ton.

If the value per short ton of 2,000 lb (used in North America) is required, the weight of the assay-ton is 29.166 g, since there are 29,166 Troy oz in 2,000 lb avoirdupois. (p. 441)

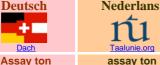
Smith

The <mark>Assay-Ton</mark> System —To facilitate calculation a system of weights, known as the "assay-ton system" (abbreviated A.T.) has been devised, which is a combination of the Troy and Avoirdupois systems with the French metrical system. The unit of the system is the "assay-ton", which for the English system (2,240 lb to the ton) is 32.6666 g. (p. 59)

Assay ton (abbreviation 'AT') is not a unit of measurement, but a standard quantity used in assaying ores of precious metals; it is 29+1/6 grams (short assay ton) or 32+2/3 grams (long assay ton), the amount which bears the same ratio to a milligram as a short or long ton bears to a troy Wikipedia ounce. In other words, the number of milligrams of a particular metal found in a sample of this size gives the number of troy ounces contained in a short or long ton of ore.















SECTIONS

BIBLIOGRAPHY:

TEXTBOOKS

► INTERNET

ARCHIVE

▶I.A.

FIRE ASSAY

ICONS

ASSAYERS' ESSENTIAL MUST-READ BOOKS

DETAILS

4. REFERENCE BOOKS - Free download ► INTERNET ARCHIVE (I.A.)

♦ 4.1 "A Textbook on Fire Assay" ♦ 4.2 "Fire Assaying" Orson C. Shepard & Waldemar F. Dietrich

♦ 4.3 "A Manual on Fire Assaying" ♦ 4.4 "The Sampling and Assay of the Precious Metals"

♦ 4.5 "Metallurgy of Gold"

♦ 4.6 "The Precious Metals: comprising Gold, Silver and Platinum" ♦ 4.7 "A Text Book of Assaving"

♦ 4.8 "Introduction to Applied Fire Assay Theory"

Thomas K. Rose C. Beringer & J. J. Beringer

Don Juergenson & Thomas J. Gilbert ♦ 4.9 "Fundamentals of analysis of gold, silver & platinum group metals" Corby G. Anderson

Charles Herman Fulton

▶ Brewster Kahle

Edward E. Bugbee

Ernest Alfred Smith

Thomas K. Rose

Bugbee Shepard **Fulton Smith** Rose1 Rose2 Berina. Gilbert Anderson

LINKS

CUPELLATION PRODUCTS: >Cupels >Blocks >Tools >Consumables - FIRE ASSAY PRODUCTS: >Crucibles >Tools / Accessories >Parting / Annealing MULTILINGUAL TERMS: >Cupellation >Fire Assay >Reagents >Other Methods >Metals >Context - INDEX: >Programme









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ESSENTIAL FIRE ASSAY TERMS

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CITATIONS FROM AUTHORITATIVE TEXTBOOKS & REFERENCE MANUALS

"BLANK ASSAY" - "BLANK TEST"

AUTHOR

CONTEXT

The best reagents obtainable for assaying contain some gold and silver. As a rule, the amount is so small that it may be neglected, but this Shepard point should always be determined by "blank" assays of the reagents. Each new lot of reagents should be tested by a blank assay, made with assay silica that is treated as a quartz ore (p. 228).

Smith

The fluxes and other reagents commonly employed in dry assaying do not contain silver or gold, but if they are left exposed in the laboratory they sometimes become accidentally (and perhaps purposely) contaminated or "salted" with material containing gold and silver values.

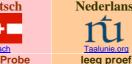
A blank assay (that is, a charge in which the ore is omitted) of the reagents will readily determine whether any precious metal is present. (p. 145 -146)

Smith

The mercury used must be free from gold and silver. As the commercial mercury may contain an appreciable amount of gold and silver it is desirable to run a blank assay on 50 cm³ to determine the amount present. (p. 239)











DETAILS



prueba en blanco



SECTIONS

ICONS

▶ Brewster Kahle

Charles Herman Fulton

Edward E. Bugbee

Ernest Alfred Smith

Thomas K. Rose

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► INTERNET ARCHIVE ►I.A.



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Orson C. Shepard & Waldemar F. Dietrich

Bugbee Shepard **Fulton** Smith Rose1 Rose2 Berina. Gilbert Anderson

LINKS

CONTEXT: ► Assay Ton ► Blank ► Steps ► Feathers ► Matte/Speiss ► Colours ► Spritting ► Sprout ► Surcharge ► Inquart ► Hallmark ► Cupels TERMS: ▶ Cupellation ▶ Fire Assay ▶ Reagents ▶ Other Methods ▶ Metals - SHEETS: ▶ Cupels ▶ Crucibles - INDEX: ▶ Programme







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ESSENTIAL FIRE ASSAY TERMS

Terms03_Cup_Steps.doc



FLUXING

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CITATIONS FROM AUTHORITATIVE TEXTBOOKS & REFERENCE MANUALS



CHARGE

OPENING UNCOVERING

DRIVING

PLAY COLOURS

BRIGH-

FINISH

CUPELLATION STEPS:

"DRIVING" - "FREEZING" - "OPENING" ("UNCOVERING")

AUTHOR

CONTEXT When all is ready the buttons are placed carefully in the cupels and the muffle door again closed. If the cupels are thoroughly heated, the lead will

melt at once and become covered with a dark scum. If the temperature of the muffle is correct this will disappear in the course of a minute or two when the molten lead will become bright. The assays are then said to have opened up or "un This signifies that the lead has begun to oxidize rapidly, raising the temperature of the molten alloy considerably above that of its surroundings,

whence it appears bright. It assumes a convex surface, and molten patches of litharge passing down over this surface give it a lustrous appearance. It is then said to "drive." (p. 93) If the temperature becomes too low for the cupel to absorb the litharge, the crystals begin to form all around and close to the lead in the cupel, and soon a pool of molten litharge is seen forming all around the annular space between the lead and the cupel.

If the temperature of the cupel is not quickly raised, this pool increases in size and soon entirely covers the lead and then solidifies. When this Bugbee occurs the button is said to have "frozen," although the lead itself may be liquid underneath. Frozen assays should be rejected as the results obtained from them, by again bringing to a driving temperature, are usually low.

If the freezing is noticed at the start, it may be arrested by quickly raising the temperature of the cupel in some way, i. e., by taking away the coolers, closing the door to the muffle, opening the draft, putting a hot piece of coke in front of the cupel, etc. (p. 94)

Keep the door to the muffle closed and when the cupel is red throughout and heated to about 850°C place the packet of lead and silver carefully in the cupel and close the door to the muffle so that the lead will fuse as quickly as possible. **Bugbee** As soon as the assay begins to "drive," note the time, open the door of the muffle and lower the temperature of the cupel by checking the fire and

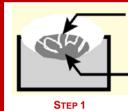
by placing cold scorifiers, etc., around it. (p. 97) All assayers agree that the best results are obtained by having a hot start, a cold drive, and a higher heat again at the finish. (p. 98) **Bugbee** It was found that before "driving" (i.e. rapid oxidation of the lead) commenced, the temperature of the molten lead rose to 900° C or above. (p. 164) Smith

Fulton If the button weighs from 15 to 20 g, as it should, it will take 25 or 30 minutes to finish the cupellation, that is, to drive off the lead. (p. 34) When the lead button is put into the hot cupel, the lead melts (326° C) and is covered by a gray-black scum. If the lead button is practically pure, as it should be, this black scum disappears when the lead reaches a temperature of 850° C. This is called the "opening up" or "uncovering" of the lead

Fulton The molten lead then appears bright, begins to "drive," and active and rapid oxidation commences.

Lead buttons should uncover as soon as possible in the muffle. If other and more difficultly fusible metals, such as Cu, Fe, etc., are present, the temperature of uncovering is higher and the temperature required for cupellation is higher. These foreign metals should, however, as a general rule, be absent. (p. 80)

CUPELLATION FLOWCHART STEPS EXPLAINED:



CLOSED LEAD

JUST BEFORE OPENING

STEP 3

FEATHERS ON A CUPEL

Dull, cracked and crusty outer surface of solid lead oxide

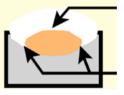
Molten lead interior

"Introduction to Applied Fire Assay (p. 55)

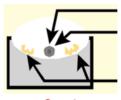
Molten lead surface continues to shine with an intense glow

Solid yellow lead oxide crystals called "feathers" form a small

distance away from molten button Lead oxide continues to be absorbed into cupel (p. 56)



STEP 2 AN OPEN DRIVING LEAD



STEP 4 FINISH, **CUPELLATION COMPLETE**

Molten lead surface is shining with intense orange-red glow

Molten lead oxide forms on the molten lead, slides off, and gets absorbed into the cupel

Molten metals don't "wet" cupel surface (p. 55)

Incandescent glow ends

released

A round, solid *doré* forms after fblink" flash occurs, as the latent heat of the molten metal is

"Feathers" remain well clear of the doré, about half-way up the cupel cavity' (p. 56)

"BLICK" - "BLINK" - "BRIGHTENING" - "FLASH" - "SCINTILLATION"

AUTHOR

Gilbert

CONTEXT If now the temperature of the muffle is below that of the melting-point of silver (962° C), or below that of the gold-silver alloy constituting the bead, or if the cupel be withdrawn from the furnace, the "blick" or "brightening" or "flash" of the bead takes place; i.e., the bead suddenly becomes very bright, at the moment of solidification, owing to the release of the latent heat of fusion, which raises the temperature of the bead very much for a short

Fulton The bead has been in a state of surfusion, i.e., in a state of fusion below its true freezing-point, toward the last of the cupelling operation; and if it be lightly jarred or the temperature allowed to drop still lower (by taking it out of the muffle), it suddenly congeals and assumes a state normal (solid) to the temperature existing. The release of the latent heat, raising the temperature of the bead, causes the brightening.

The "brightening" of very small beads is rarely noticeable. (p. 82) **Fulton** It is an old saying amongst assayers that "a cool drive and a hot blick" are essential to a good cupellation. (p. 86)

Fulton Beads containing more platinum than 1 in 16 will not blick or flash. (p. 187)

If the bead was in a state of surfusion at the finish and consisted of nearly pure gold and silver it would solidify upon further cooling with the emission of a flash of light (known as the "blick" or "flash"). This flash is due to the sudden release of the latent heat of fusion of the alloy at the moment of solidification, which momentarily raises the temperature considerably. The blick is seldom perceptible in beads larger than 700 mg. Small amounts of lead or copper in the bead diminish the intensity of the blick Shepard

All the platinum group of metals, with the exception of platinum and palladium, suppress the blick entirely. If the blick is observable, it is a useful guide to the completion of cupellation, but the assayer need not waste time trying to observe the blick, as the end of cupellation is easily ascertained by other observations. (p. 63) If the cupel is withdrawn from the furnace, the button becomes very bright at the moment of solidification and is said to "flash", "brighten" or "blick".

cooling globule to its melting-point. **Smith** The flashing is much more noticeable with gold than with silver buttons. Molten gold has a peculiar green colour which is easy to recognise and just after solidifying it glows beautifully, even with very small buttons. With very small silver buttons the flashing is rarely noticeable. The flashing is prevented if the buttons contain metals of the platinum group. (p. 160)

This "flashing" of the buttons of gold or silver is due to the evolution of the latent heat of fusion or "recalescence", which momentarily reheats the

Kühl getrieben, heißer Blick, ist des Probierers Meisterstück (p.161) Probierbuch Kalt getrieben und heiss geblick ist im probiren ein Meisterstück Cool at driving, hot at 'blick', this is the assayer's master trick. Kalt <mark>getrieben</mark> und heiss <mark>Blick</mark> ist der Probierer Meisterstück **Neuss**



SECTIONS FIRE ASSAY

TEXTBOOKS

►I.A.



ICONS

ASSAYERS' ESSENTIAL

MUST-READ BOOKS

♦ 4.1 "A Textbook on Fire Assay" ♦ 4.2 "Fire Assaying"

♦ 4.3 "A Manual on Fire Assaying"

♦ 4.4 "The Sampling and Assay of the Precious Metals"

♦ 4.5 "Metallurgy of Gold"

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♦ 4.7 "A Text Book of Assaying"
C

C. Beringer & J. J. Beringer Don Juergenson & Thomas J. Gilbert ♦ 4.8 "Introduction to Applied Fire Assay Theory" ◊ 4.9 "Fundamentals of analysis of gold, silver & platinum group metals" Corby G. Anderson

LINKS **Bugbee**

▶ Brewster Kahle

Edward E. Bugbee

Ernest Alfred Smith

Thomas K. Rose

Thomas K. Rose

Charles Herman Fulton

Orson C. Shepard & Waldemar F. Dietrich

Shepard **Fulton** Smith Rose1 Rose2 Bering. Gilbert **Anderson**

• 4. REFERENCE BOOKS - Free download ▶ INTERNET ARCHIVE (I.A.)









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ESSENTIAL FIRE ASSAY TERMS

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DRIVING

PLAY COLOURS TENING Виск

FINISH

"FEATHERS"

AUTHOR Fulton

CONTEXT ... crystals of litharge (feathers) form on the side of the cupel toward the muffle mouth. If the temperature is too low for the cupel to successfully absorb practically all of the PbO, these feathers form low down in the cupel. When the temperature is about right, they form near the upper rim of the cupel. It is, however, to be noted that the draft through the muffle influences the formation of feather litharge; i.e., if the draft is strong, feathers

will form, although the temperature is somewhat above 820° C. (p. 82) eathers," will appear around the upper edge of a bone-ash cupel. This is due to the condensation of that small part of the litharge which is volatilized at the surface of the lead. If the temperature is too high the volatilized litharge is carried away in the furnace gases or is deposited on cooler projecting surfaces in the path of the air stream as it passes through the furnace.

If the air draft is too strong, feathers may appear only on the side of the cupel toward the draft, or may not form.

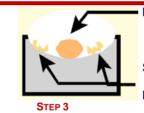
As cupellation proceeds and the lead button becomes smaller, concentric rings of feathers are deposited within the original ring, but at all times the temperature should be high enough so that there is a clear area between the button and the feathers, otherwise there is danger that a Shepard pool of litharge will form immediately around the button, which will instantly prevent further absorption of litharge by the cupel, and the litharge will soon completely cover the lead and solidify, which will cause the button to freeze.

Even though the assayer fails to note the encroachment of feathers toward the button, or the decrease in color temperature of the lead, the onset of freezing is plainly evident in the oily appearance of the ring of molten litharge at the outer periphery of the button, and the temperature should be raised immediately to avoid freezing.

Individual cupels that show signs of incipient freezing may be saved by placing a hot cupel or brick near or over them. (p. 59)

The formation of feathers of litharge can be observed readily with bone-ash or bone-ash-cement cupels and serves to indicate proper driving Shepard temperature, but when copious feath ers form on magnesia cupels the temperature is dangerously near the freezing point. (p. 66)

Gilbert



Molten lead surface continues to shine with an intense glow

Solid yellow lead oxide crystals called "feathers" form a small distance away from molten button

Lead oxide continues to be absorbed into cupel

FEATHERS ON A CUPEL "Introduction to Applied Fire Assay Theory" (p. 56)

"Feathers" are crystals of solid litharge sublimed from the vapour and deposited on the rim of the cupel, which is invariably cooler than the Smith molten lead on the cupel. It is stated by Fulton that they will not form above 820° C. (p. 162)

When cupellation of silver is carried on under these conditions the temperature should not, according to Fulton, be above 820° C, in which case crystals of litharge (feathers) form on the side of the cupel towards the muffle mouth.

If the temperature is too low for the cupel successfully to absorb practically all the litharge, these feathers form low down in the cupel. When the temperature is correct, they form near the upper rim of the cupel. It is, however, to be noted that the draught through the muffle influences the formation of feather litharge; i.e. if the draught is strong, feathers will form, although the temperature is somewhat above 820° C. (p.

Smith

Smith

The formation of "feathers" is generally accompanied by a sluggish, heavy movement of the fumes, which fall in the muffle. Beyond the fact that it shows that the muffle has not been too hot, it is doubtful whether there is any advantage in cupelling at a temperature sufficiently low to permit of the formation of feathers. (p. 163)



Deutsch Dach

Feder (Bleioxid)

Nederlans

veer (litharge)

Francais Francophonie.org

plumes (litharge)

Italiano penne (litargirio)

Castellano

plumas (litargirio)

▶ Brewster Kahle

Charles Herman Fulton

Ernest Alfred Smith

Thomas K. Rose

Thomas K. Rose

перья (окись свинца)

Русский

BIBLIOGRAPHY:

SECTIONS **ICONS** FIRE ASSAY

Техтвоокѕ

► INTERNET ARCHIVE ►I.A.



ASSAYERS' ESSENTIAL MUST-READ BOOKS

DETAILS

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Rose1 Rose2 Bering. Gilbert Anderson

LINKS

Bugbee

Shepard

Fulton

Smith





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2.05

ESSENTIAL FIRE ASSAY TERMS

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SAMPLE Preparation

FLUXING

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CITATIONS FROM AUTHORITATIVE TEXTBOOKS & REFERENCE MANUALS

"MATTE" ("REGULUS") - "SPEISS"

CONTEXT **A**UTHOR

E is an artificial sulphide of one or more of the metals, formed in the dry way. In assaying it is most often encountered in the niter fusion of sulphide ores when the charge is too acid. It is found lying just above the lead button. It is usually blue-gray in color, approaching galena in composition and very brittle. It may form a layer of considerable thickness, or may appear simply as a granular coating on the upper surface of the lead button. This matte always carries some of the gold and silver and, as it is brittle, it is usually broken off and lost in the slag in the cleaning of the lead button. The student should examine the lead button as soon as it is broken from the slag and if any matte is found, he may be certain that his charge or furnace manipulations are wrong.

ISS is an artificial, metallic arsenide or antimonide formed in smelting operations. As obtained in the fire assay, it is usually an arsenide of iron Budbee Budbee approaching the composition of Fe₅As. Occasionally the iron may be replaced by nickel or cobalt. The antimony speiss is very rare. In assaying, speiss is obtained when the iron method is used on ores containing arsenic. It is a hard, fairly tough, tin-white substance found directly on top of the lead and usually adhering tenaciously to it.

If only a small amount of arsenic is present in the ore, the speiss will appear as a little button lying on top of the lead; if much arsenic is present, the s will form a layer entirely covering the lead. It carries some gold and silver. If only a gram or so in weight, it may be put into the cupel with the lead and will be oxidized there, giving up its precious metal values to the lead bath. A large amount of speiss is very hard to deal with as it is difficult to scorify. The best way is to assay again, by some other method. (p. 15)

Slags for Pure Ores. - When an ore contains so large a proportion of sulphide minerals that it is necessary to add niter to prevent the reduction of too much lead, it will be found that the charges recommended for Class 1 ores will not allow a satisfactory decomposition of the ore. Instead of two products, slag and lead, a third intermediate product, matte, is often obtained as the result of the fusion. This amounts to an incomplete decomposition of the ore and as matte is a good collector of precious metals its presence is a sure indication of low results.

A matte is much less likely to be formed, however, with a less acid charge and it has been found best, therefore, to make a slag approaching a monosilicate for all sulphide ores, as by this means more uniformly satisfactory results are obtained. (p. 175) - In the melts used in assaying, smelting, and fire refining there is the possibility of producing a number of separate liquids that are not

miscible with each other and, therefore, segregate into separate layers called "phases". Any or all of the phases — metal, speiss, matte, slag, and molten alkaline salts — may be formed. The metal phase has the highest density and forms the bottom layer; the other phases separate above the – metal. speiss, matte, slag, and metal phase according to their densities, which are usually in the order given above.

The speiss and matte phases are avoided in fire assaying by proper conduct of the assay. Speiss consists of the arsenides and antimonides of iron, cobalt, nickel, or copper; matte is a mixture of fused sulfides, usually of iron and copper. Speiss is heavier than matte but lighter than lead; hence Shepard it forms a layer between the lead and the matte, if all three are present.

The slag phase, consisting of metal oxides and silica or borax glass, lies above the matte layer or, if matte and speiss are absent — as should be the case in an assay fusion — the slag rests directly upon the lead. (p. 86)

the case in an assay fusion — the slag rests directly upon the lead. (p. 86)

Whether a metal appears in the metal layer or in the slag depends upon whether or not it is combined with oxygen. The metal oxides are quite soluble in the slag and, in general, are insoluble in the metal. Consequently, when two different metals are present, if one can be oxidized to metal oxide it will go into the slag layer, while if at the same time the other metal can be prevented from oxidizing it will appear in the metal layer, provided

no speiss or matte is formed. This principle forms the basis of the separations made in fire assaying, smelting, and fire refining. (p. 88)

In the presence of copper, speiss will form in the assay fusion unless the charge is proportioned with sufficient excess of litharge and soda to keep the copper as well as the arsenic and antimony oxidized.

Uncontrolled reduction methods should never be used for assaying materials containing large amounts of arsenic or antimony. The excess reduction used in uncontrolled reduction methods will reduce arsenic and antimony, as well as some of the speiss-forming metals, so that speiss is almost certain to form.

When a speiss phase forms, it is found as a brittle substance just above the lead button. It can be distinguished from matte by its bright metallic **Shepard** luster.

The danger of loss of precious metals in the presence of speiss occurs primarily through loss of small fragments of speiss when separating the lead

button from the slag; but even though all the speiss could be recovered, it cannot be cupeled satisfactorily

Artificial sulfides of the heavy metals are called "matte." In general, mattes are almost completely insoluble in slag and have only slight solubility in metal or speiss phases, but they are soluble in molten alkaline salts such as sodium carbonate and, particularly, sodium sulfide. (p. 111-112)

Smith

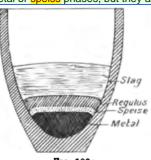


Fig. 1004



In addition to slag and metallic lead, other products may be obtained as the result of the fusion of an ore according to the constituents present. If metallic *sulphides* are present, an artificial sulphide or *regulus* (sometimes termed *matte*) may be formed and if the ore contains arsenical minerals, a compound of a metal or metals with arsenic, termed a eiss, mav result.

Assuming these substances to be present in a charge, they would separate according to their densities, when the fused mass solidified, in the order shown in fig. 100A.

At the bottom of the crucible a button of lead would be found; above this a thin layer of

s; then a regulus, next a slag, and, in special cases, on the top of this a layer of more fluid slag consisting usually of fusible alkaline chlorides and sulphates.

A regulus (also termed matte), which is a compound of one or more metals with

sulphur, is a yellowish-grey, metallic-looking mass, usually brittle and often crystalline. A ss is usually hard, brittle and greyish-white in appearance. (p. 127-128)



Deutsch Matte, Stein, Lech

Speise

Nederlans matte speiss

Francais Francophonie. matte speiss

Italiano matte

speiss

DETAILS

mata

Castellano

speiss

► Brewster Kahle Edward E. Bugbee

Ernest Alfred Smith

Charles Herman Fulton

Русский штейн шпейз

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• 4. REFERENCE BOOKS - Free download ► INTERNET ARCHIVE (I.A.) ♦ 4.1 "A Textbook on Fire Assay"

◊ 4.2 "Fire Assaying"

♦ 4.3 "A Manual on Fire Assaying"
 ♦ 4.4 "The Sampling and Assay of the Precious Metals"

♦ 4.5 "Metallurgy of Gold"
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Orson C. Shepard & Waldemar F. Dietrich

Shepard **Fulton Smith** Rose1 Rose2 Bering. Gilbert Anderson

LINKS

Bugbee









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OPENING UNCOVERING

DRIVING

COLOURS

TENING

FINISH

"PLAY OF COLOURS" - "BRIGHTENING"

AUTHOR CONTEXT

As the alloy becomes richer in silver it becomes more and more rounded in shape and shining drops of litharge appear and move about on its

As the last of the lead goes off, these drops disappear, the fused litharge covering becomes very thin and, being of variable thickness, gives an Bugbee effect of interference of light, so that the bead appears to revolve and presents a succession of rainbow colors.

This phenomenon is termed the "play of colors."

The colors disappear shortly, the bead becomes dull and after a few seconds appears bright and silvery.

This last change is called the "brightening." . (p. 95)

The visual phenomena that appear near, and at the finish of cupellation are of considerable aid to the assayer.

Since both silver and gold have a higher surface tension than lead the button becomes more rounded as the percentage of lead decreases; this effect becomes pronounced as the proportion of precious metals increases beyond 50%.

When the cupellation of large beads approaches completion, oily-appearing drops of litharge can be seen to collect on the surface of the bead. These particles appear to move, and the movement becomes increasingly more rapid until just before the finish, when the molten litharge forms

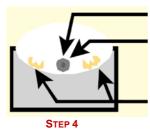
Shepard a thin film of variable thickness and creates interference colors.

The rainbow color bands move swiftly over the surface of the button and give the illusion that the button is revolving about a shifting axis. This is known as the "play of colors" and is strikingly developed with large beads.

When the last trace of lead has been removed from the bead the play of colors disappears and the bead becomes dull for a brief period, after which it acquires a normal metallic luster.

This change of luster is known as "brightening" and is not always observable. (p. 62)

Gilbert



Litharge flowing on surface of bead gets thinner, creating a fast moving iridescent luminous film, this phenomena is called Play of Colours Incandescent glow ends

A round, solid doré bead forms after "blink" flash occurs, as the latent heat of the molten metal is released

"Feathers" remain well clear of the doré, about half-way up the cupel cavity"

"Introduction to Applied Fire Assay Theory" (p. 56)

CUPELLATION COMPLETE ... just as the last of the lead oxidizes (play of colors). (p. 95)

FINISH,

Fulton

The drops of litharge which in the earlier stages flow steadily from the surface of the alloy, thin off later to a luminous film. **Bugbee** At the end this film appears in commotion, then presents a brilliant play of colours, and, with a sudden extinction, the operation is finished. The metal again glows for an instant whilst becoming solid. (p. 99)



Farbenspiel /

Schillern









iridiscencia



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Ernest Alfred Smith

Thomas K. Rose

Thomas K. Rose

▼ **Bugbee Shepard** <u>Fulton</u> **Smith** Rose1 Rose2 Bering. Gilbert

Anderson

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"SPIT" - "SPITTING"

AT BEGINNING OF CUPELLATION

AUTHOR CONTEXT Carbon is an undesirable constituent of cupels as it reacts with the lead oxide formed giving off CO and CO2 which may cause a loss of the Bugbee molten alloy due to spitting. (p. 90) The presence of CaO₃ is very undesirable in bone-ash for cupels, as it begins to give off CO₂ at 800° C, about the temperature of the beginning **Fulton** of cupellation, causing a serious spitting of the lead button, which entails a loss of the precious metals. (p. 77) If the buttons were placed into the cold cupel, the lead would melt before all the remaining moisture is expelled, which would then pass up violently through the molten lead, causing what is termed "spitting," i.e., the projection of small lead particles, carrying gold and silver from the cupel. Some cupels, made from bone-ash containing CaCO₃, will commence to spit after the cupellation has proceeded for some time and the temperature has risen to above 800° C. **Fulton** This can be stopped by pulling the cupel to the cooler (front) part of the muffle, although the cupellation, after spitting, is to be considered unreliable. When a piece of wood or coal is placed in the muffle to "open up" lead buttons, the cupels absorb gases at times, which later on, when the temperature rises, are again expelled, with a spitting of the lead. (p. 80) The presence of organic matter and of carbonates, nitrates, and other salts that decompose at cupellation temperature (850 to 900°C) or lower **Shepard** is undesirable, as the evolution of gases during cupellation causes loss by "spitting" of the lead. (p. 49) Whatever free moisture may be present in an air-dried cupel is expelled when the cupels are preheated in the muffle just before using, a practice Shepard which is necessary in all cases in order to remove combined water, CO₂, and other volatile matter that would cause spitting and to avoid delayed opening of the buttons if placed in cold cupels.(p. 54) Before cupellation the set of cupels should be charged into the furnace and heated at 850 to 900°C for 10 min., with the draft slightly open to provide an oxidizing atmosphere. This will drive off free and combined water, organic matter, carbon dioxide, and other volatile constituents that

Shepard would otherwise rise through the lead during cupellation and cause the ejection of particles of lead.

This phenomenon is known as "spitting" and is a source of loss to the assay in question, as well as the cause of salting other samples into which the globules of lead may fall. (p. 56)

Ores that decrepitate violently are troublesome in scorification because of "spitting," which is the violent projection of small particles of lead from the scorifier.

Shepard

... Place the scorifier into a muffle at 500 to 600°C, close the door and muffle draft, and heat for 2 or 3 min. until the lead is melted and danger of decrepitation or spitting is past. (p. 166)

The bundle of lead foil containing the residue is frequently cupelled directly as a lead button from an ore fusion. Spitting, that is, the ejection of small droplets of lead into the air, is likely to take place soon after cupellation starts. This is caused by the action of the salts in the residue. Loss due to spitting is not so serious as one might expect; nevertheless, it should be avoided when high accuracy is desired. Spitting can be avoided by a brief preliminary scorification. (p. 196)











DETAILS



▶ Brewster Kahle

Edward E. Bugbee

Ernest Alfred Smith

Thomas K. Rose

Charles Herman Fulton



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Rose

Fulton

Beringer

Smith

Smith

Smith

sprouting

vegetation



WARE-

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"SPIT" - "SPITTING" - "SPROUT" - "SPROUTING" - "SPURTING" - "VEGETATION"

AT END OF CUPELLATION

AUTHOR CONTEXT

Molten silver absorbs about twenty-two volumes of oxygen from the air, and the gas is given off again during solidification, usually with much vigour. The solidified crust of silver is generally broken and blistered, and particles of metal are projected by the gas disengaged from the interior Rose (the so-called "spitting" or "sprouting").

If no copper had been added, the disengagement of oxygen at the moment of solidification would cause the button to spurt or "spit," and the assay would be spoilt by the loss of some metal

... the furnace is allowed to cool down slowly and evenly. If the molten beads were removed before solidification, spitting would result, but as Rose soon as they have solidified they are withdrawn from the furnace, .. Platinum is not affected by heating in dry or moist air. When molten it absorbs oxygen which is given off on cooling, sometimes with sufficient Rose

rapidity to cause the metal to spit like silver. Liquid silver has the peculiarity of dissolving a large volume of oxygen, which is expelled upon solidification. The solidification of large beads starts at the surface. When the center of silver beads solidifies, the oxygen is sometimes expelled violently, spewing forth some of the interior silver and forming a cauliflower-like growth on the bead. This mechanism is known as "sprouting.

<mark>ut</mark>ed beads should be rejected if there is any reason to believe that any particles of metal have been lost.

To avoid sprouting, all large beads should be cooled slowly, either by leaving them in the furnace after the blick, and until certain that they have cooled sufficiently, or by moving them to a slightly cooler zone in the furnace and then covering them with a very hot cupel before they have completely solidified.

The hot cupel melts the outer crust and allows the bead to solidify slowly, so that the oxygen can escape from the interior without violence. When the bead contains more than one-third of its weight in gold, it does not sprout; hence beads known to be of this composition may be removed from the furnace as rapidly as desired. Sprouting of silver beads is considered a sign of purity, but this indication is of no practical value to the assayer, as sprouting should always be

avoided. (p. 63) Bugbee states that as little as 0.004% of rhodium in silver beads causes a distinct crystallization, which is more apparent at 0.01% Rh, and that

Shepard 0.03% Rh causes unavoidable sprouting of silver beads. (p. 71) . there is no danger of <mark>sprouting</mark> if the silver-gold ratio is less than 3 to 1, and if copper is present. (p. 182) Shepard

Bugbee outing, however, is considered proof of the absence of all but traces of impurities. (pag. 114)

Instead of attempting to prevent sprouting by covering with a hot cupel, the student may try the following little-known method, first described by Aaron. After brightening, the cupel is drawn to the front of the muffle and gently tapped on one side with the tongs. <u>Bugbee</u> At the instant when the bead ceases to vibrate in response to the taps, by which is indicated the beginning of solidification, it is pushed back into

the hottest part of the muffle and left for about a minute. After this it may be entirely withdrawn and will not sprout, being solid all through, as shown by a "dimple" in its surface, caused by contraction. (p. 228) Silver beads after cupellation, and at the moment of solidification, also "sprout". According to Gay-Lussac molten silver dissolves 22 times its volume of oxygen, at the freezing-point. Later researches prove this practically

correct. At 1020° C molten silver will hold 19.5 volumes of oxygen (at 760 mm and 0° C) and at the melting-point somewhat more. For any given temperature the oxygen dissolved is proportional to the square root of the oxygen pressure. In air at 760 mm pressure the oxygen has a partial pressure of 150 mm and the volume of oxygen dissolved by molten silver under assay conditions is 9.65 volumes at the freezing-point of silver The oxygen is dissolved either as monatomic oxygen or as silver oxide (Ag₂O), in dilute solution. It is probable that this silver oxide, not being

soluble in solid silver is dissociated with explosive violence, with the liberation of oxygen, when the silver solidifies.

This oxygen, suddenly expelled when the bead solidifies, causes a cauliflower-like growth on the bead. Small particles of silver may even be projected from it and cause a serious loss. When gold is present in the silver bead to the extent of 33 %, or more, sprouting does not take place. Silver beads containing small quantities of Pb, Cu, Zn, Bi, etc., will not sprout, so that if a button does sprout it is a sign of purity.

Buttons below 5 mg in weight do not sprout readily; large buttons, however, do. Sprouting can be prevented by slow cooling in the muffle, or by having ready a hot cupel which can be set, inverted, over the one holding the bead, and withdrawing both from the muffle, thus cooling the bead <mark>ut</mark>ed beads are to be rejected as an assay. (p. 83) Molten silver dissolves oxygen from the air and gives it off on solidifying; the escape of the gas on sudden cooling is violent and, by throwing off

particles of the metal, may cause loss. This is called "vegetation" or "spirting. The silver is apparently solid when spirting takes place; the crust breaks suddenly and some of the metal is forced out. The evil is best guarded against by slow cooling and avoiding draughts.

Beringer With large buttons of silver precautions should never be omitted. One plan is to allow the cupels to cool in the muffle itself, the mouth being

closed with hot charcoal. Another is to cover the cupel with another cupel previously heated to redness; in this case the silver cools between two hot cupels, and, of course, cools slowly. A third plan is to withdraw the cupel to the door of the muffle, holding it until it begins to get solid and then immediately to put it back into the hotter part of the muffle (p. 100). But the danger of spirting decreases as the proportion of gold becomes greater, and disappears when the gold is much over 30%.

the muffle and allowing the furnace to cool down somewhat before withdrawing the cupels. (p. 144). Silver when molten absorbs oxygen from the air and gives it off suddenly when solidifying; causing a cauliflower-like growth on the surface of the button and particles of silver may even be ejected out of the cupel and cause serious loss. This "spirtting", "spurting" or "vegetation" as it is termed does not take place readily with silver buttons weighing less than about 5 mg, but all buttons above this weight are very liable to spit. Spitting does not take place if gold is present in the silver button to the extent of 33 % or more (Levol), as the solubility of oxygen in silver is lowered by alloying

Nevertheless it is well to let such buttons become solid undisturbed and protected from draughts in the body of the muffle. This means closing

with gold. If the button is small the cupel may be withdrawn from the muffle as soon as cupellation is finished without risk of "spitting", but with large buttons precautions must be taken. g can be prevented by slow cooling and in practice the following methods are usually adopted. The cupels are allowed to cool in the muffle itself, the door being closed; or another cupel, previously heated to redness, is inverted over the cupel containing the button and both carefully

withdrawn from the muffle, thus cooling the button slowly between two hot cupels; or the cupel may be withdrawn very gradually towards the door of the muffle. Cooling in the muffle is invariably adopted with a large batch of assays, as, in addition to the danger of upsetting a cupel, it is inconvenient to

handle a large number of hot cupels Buttons that have "spitted" must be rejected and the assays repeated. (p. 160) Vegetation

The oxygen evolved often bursts through the outer crust of solidified metal with considerable violence, ejecting portions of the still liquid silver as irregular excrescences constituting the phenomenon known as the "spurting", spitting" or <mark>vegetation</mark> of silver.

This may be prevented by sprinkling charcoal powder on the melted metal. The presence of small quantities of copper also prevents the spurting of silver.

Silver alloyed with as much as one-third of its weight of gold still retains the power of absorbing oxygen and

spurting on solidification, but larger proportions of gold prevent the action (Percy).

The cupels retain the heat longer than the muffle and if the cooling is too rapid, the buttons will solidify on the exposed surface while the interior

is kept molten by contact with the hot cupel and when the button solidifies, spitting is very liable to take place. If, however, the cupel is slowly cooled from below, the under surface of the button will solidify first and the dissolved oxygen escape before the

silver solidifies as a whole. (p. 184). The copper is added as the small quantity that remains in the button tends to prevent "spitting" or vegetating after cupellation and also to **Smith**

increase the malleability of the button for rolling. (p. 267) To avoid losses by "spitting", etc., most assayers allow the assays to remain in the furnace until the buttons have solidified. To effect this the **Smith** muffle-door is closed and the temperature lowered. The buttons when "set" (solidified) should show a depression on the top and no "vegetation"



spruitje *i*

scheut

sprießen, Sproß,

Blumenkohl





Castellano



Русский



arborescence









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"SURCHARGE" - "CHECK" - "PROOF" - "STANDARD"

AUTHOR CONTEXT Surcharge — Net Losses and Gains -The net sum of the (...) losses and gains incurred in the various operations is called the "surcharge" since, in the assay of high-grade bullion, the silver retained is usually in excess of the gold lost, so that the cornet will weigh more than the gold originally present in the assay piece; if the reverse is the case, the result is considered by some assayers to be less accurate. When there is a gain in weight it is referred to as a "plus surcharge", while a loss in weight is designated a "minus sui Smith With material rich in gold, the silver retained more than compensates for the loss, and, as just stated, the surcharge is positive; but with a decrease in the proportion of gold the loss is greater and the surcharge is negative. Thus the surcharge will usually amount to about 0 (nil) for bullion of about 700 to 800 fine; above that there will be a "plus surcharge" and below that a "minus surcharge" The surcharge is reported in parts per 1000 in the same way as the fineness of bullion. (p. 276) .. certain losses and gains (the sum of which is called the surcharge) take place during the cupellation and parting of gold alloys, the losses being due to (1) absorption by the cupel, (2) volatilisation and (3) solution in the acid and the gains to the retention of silver by the gold cornet. **Smith** The amount of the surcharge varies with the conditions of working and the composition of the alloy, the general experience being that the losses under all three heads are greater as the percentage of copper in the alloy increases. (p. 328) The total losses may or may not be counterbalanced by the silver retained by the gold after parting, which amounts to about 1 part per thousand under normal conditions of working. In practice it is found that with alloys rich in gold the silver retained by the cornet more than compensates for the various losses and the **Smith** urcharge is positive; but with alloys of low standard the losses are greater and the surcharge is negative, the cupellation loss being usually greater on account of the larger proportion of copper present, as previously pointed out. (p. 330) Since the losses and gains are dependent on so many conditions, the surcharge is best determined by the use of "checks" or "proofs" consisting of pieces of pure gold which are subjected to the various operations side by side and under identical conditions with the assay pieces. The composition of the checks is the same as the composition of the bullion being assayed in order to make the assays absolutely comparable. **Smith**

Being, therefore, of the same composition and weight and undergoing exactly the same treatment, the checks may reasonably be expected to have the same surcharge as the assays which they imitate. The checks are made up from the data obtained by a preliminary assay. (p. 276)

Consequently it is necessary to subject pieces of fine gold (called "proofs" or "checks") to the various operations side by side and under identical conditions with the assay pieces, and thus to determine the "surcharge" or net sum of the losses and gains incurred in the various operations.

The surcharge is usually positive, a proof weighing 1,000 at the beginning attaining a weight of about 1,000.5 at the end.

If there is a surcharge in the check of more than 1‰ or 2‰ in the first cupellation, showing retention of copper, the prills are re-cupelled with Rose fresh lead. In the bullion assay for gold, the algebraic sum of the errors outlined, the losses being designated minus and the gains plus, is called the

surcharge In the gold bullion assay this will vary from +0.025 %, in very pure gold bullion, to 0.25 %, in base bullion, passing to zero for a bullion about 800 fine. (p. 173)

This surcharge will usually amount to about "0" for a bullion of about 700 to 800 fine; above that there will be a "plus surcharge," and below that a "minus surcharge." The plus surcharge will be subtracted and the minus surcharge added. (p. 185) The proofs always show a slight gain in weight. The correction thus determined is termed the "surcharge," and is really the algebraic sum of all

Bugbee the gains and losses. (p. 231). The sum of the errors of an assay, which is called the surcharge, is reported in the same way. Thus a surcharge of + 0.3 means that the gold as weighed was 0.3 parts per 1000 more than the gold actually present. But a surcharge – 0.3 means that on the whole there was a loss of 0.3% in the

Speaking roughly the retained silver will vary with the weight of gold present; if one alloy contains twice as much gold as another the retained Beringer silver will be about twice as much also

On the other hand, as already explained, the cupellation loss on the poorer alloy is as much as, or even more than, with the richer one, because of the copper, &c. present. With rich gold alloys the silver more than compensates for the loss and the surcharge is positive; but with poorer alloys the loss is greater and the surcharge is negative. (p. 155).



▶I.A.

Rose

Fulton

Fulton

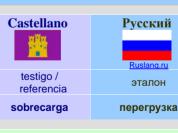


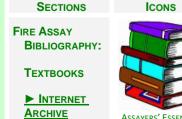
Überlastung















DETAILS







FIRE ASSAY SL - CARRER DE NOVELL, 54 - 2nd FLOOR ES-08014 BARCELONA - SPAIN - EU

WARE-

TSM Log. – B-682, CTRA. ACCÉS COSTA BRAVA km 1,4 WARE-HOUSE TSM LOG. — B-682, CTRA. ACCÉS COSTA BF ES-08389 PALAFOLLS — BCN — SPAIN — EU

Smith

Canviador"



ESSENTIAL FIRE ASSAY TERMS

Oct-2024 Terms10_Inq_Part_Touch.doc

SAMPLE SLAG ANNEALING **FLUXING FUSION** CUPELLATION PARTING Preparation Separation

MEANINGS & CLUES

CITATIONS FROM AUTHORITATIVE TEXTBOOKS & REFERENCE MANUALS

"INQUART" - "INQUARTATION"

AUTHOR CONTEXT

Inquartation. – When the bead contains too little silver to part, it is necessary to alloy it with more silver. This process is called inquartation. It originated from the custom of the old assayers of adding silver until the gold was one-quarter of the whole. They considered a ratio of 3 parts of Bugbee silver to 1 of gold to be necessary for parting. At present, in assaying gold bullion, a ratio of only 2 or 2½ parts of silver to 1 of gold is used, mainly to avoid all danger of the gold breaking up in the boiling acid.

In this case some little silver remains undissolved, even though the alloy is rolled out to about 0.01" in thickness. (p. 121)

Many assayers, when working for both gold and silver and suspecting an ore to be deficient in silver, add silver to the crucible or to the lead <u>Bugbee</u> button before cupeling, part directly and then run separate assays to determine the silver in the ore. (p. 122)

Inquartation. - The method of separating the gold from the silver in gold-silver alloys by boiling with nitric acid does not act equally well in all cases. An alloy half silver half gold, rolled to thin sheet and boiled for half an hour with nitric acid, may still retain more than two-thirds of its silver. An alloy of 1 part gold and 1.7 parts of silver gives up practically the whole of its silver under similar treatment. The gold is left in a coherent, though easily broken, sheet retaining the shape of the original alloy. The gold thus left is quite spongy and porous, so that the acid can penetrate into its innermost portions. But if the silver is in large excess in the alloy, the removal of the silver is less complete, and the residual gold, instead of holding Beringer together in a form easy to manipulate, falls to a powder which requires care and time in its treatment. The older assayers, therefore, added silver to their gold in such proportion that the alloy for parting should be one quarter gold to three quarters silver. This operation they called inqua

The modern practice is to aim at getting an alloy with 2.5 parts of silver and 1 part of gold. In gold bullion assays this proportion should be obtained with fair exactness. And in the parting of such gold buttons as are obtained in assaying ores it is well to aim at this proportion, though absolute precision is not a matter of importance. (p. 146)

The silver used for inquartation must, of course, be free from gold and is best prepared by the assayer who is to use it.— It should not be in Beringer long strips or angular pieces likely to perforate the lead in which it is folded. When wrapped in the lead it should be in the middle and should make as compact a parcel as possible. (p. 148)

quartation. – In order to ensure complete parting without breaking up of the gold, assay beads containing too little silver must be inquarted with Shepard as in the gold bulliar account for the same as much silver as gold is required, except when masses of 200 mg or more of gold are to be parted, as in the gold-bullion assay, when the ratio of silver to gold is 2:1 or 3:1. Early assayers believed that the best alloy for parting contained one-fourth gold and three-fourths silver. In fact the literal translation of the Latin word for parting, quartatio, is "fourthing". (p. 75)

Proof silver used for inquartation is sometimes stamped into discs of convenient size and weight or drawn into flat wire and pieces of the required weight cut off with gauged pliers. Some assayers consider it more convenient for weighing out to granulate the silver and use only the granules that will pass through a sieve of about 13-mesh (p. 264)

"PARTING"

AUTHOR

Parting is the separation of silver from gold by means of acid. In gold assaying nitric acid is almost exclusively used, although sulphuric acid is Bugbee usually employed for parting large lots of bullion. Nitric acid cannot be used successfully to separate silver from gold unless there is present at least three times as much silver as gold (p 118).

The idea of parting is to so manipulate that the gold will, if possible, remain in one piece.

The nitric acid for parting must be free from hydrochloric acid and chlorine in order to have no solvent action on the gold and also because any chlorides present would precipitate insoluble silver chloride on the gold. The acid strength is of great importance and the proper strength to be used **Bugbee** depends upon the composition of the alloy. The higher the ratio of silver in the alloy, the less the acid strength should be.

Great care is necessary in parting to avoid breaking up the gold and subsequently losing some of the small particles, as well as to insure complete solution of the silver. (p. 118)

"TOUCHSTONE" — "TOUCH NEEDLE" — Also ▶ Wächli & Vuilleumier articles

CONTEXT AUTHOR Assaying with the touchstone

toetsstreek

hstone testing method is a fast non-destructive screening and assaying method. The kind of precious metal and the fineness are Waarborg determined by testing the colour and chemical resistance. The materials used are touchstones, touch acids and touch needles (alloys with an **Holland** accurately established fineness of precious metal).

The advantage of the touchstone testing method is that in principle every parts of the article can be tested.

A rubbing of the item is made on a special stone, treated with acids and the resulting colour compared to references. Differences in precious <u>Wik</u>i metal content as small as 10 to 20 parts per thousand can often be established with confidence by the test. It is not indicated for use with white gold, for example, since the color variation among white gold alloys is almost unperceivable.

Jordi de 33 Mas, pus le tocs dels metalls fer sabets 33 But given that you know the touch of the metals how to make 34 prou destrament, que·s autre be no us say, 34 skilfully enough, that I don't know if there is anything you can do so well, Sant Jordi 35 e⋅z avets favt dels bons lo bon assav 35 and you have made the best amongst the good assays,

36 e com ets tals que I millor no n triets, 36 why is it that the best you didn't choose? "<u>Lo</u> 37 no us pensets vos que us ho dia per me, 37 don't imagine that I ask you this for me,

38 qu'aicest traüt no us vull far, per ma fe. 38 that in this nuisance I do not want you to meddle, for my faith.

39 Ja no metrets vostres diners menuts (tornada) 39 No longer shall you mix your little pennies

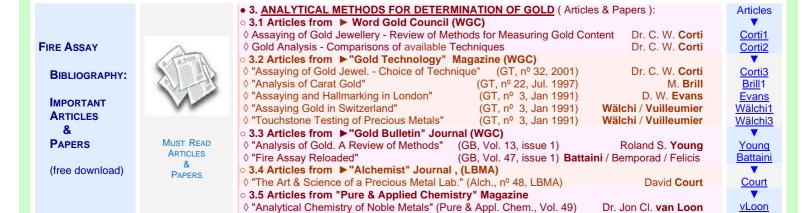
(refrain) 40 together with my florins of weight well known 40 amb mos florins de pes ben coneguts.

Nederlans Francais Italiano Castellano Русский **Dach** Taalunie.org Francophonie.org обогащение inquart (to) / inquartieren / inquart / inquarto / incuarto / vierendelen серебром / (in-)quartation (in-)quartation inquartazione incuartación (In-)Quartation (квартование) separazione, <u>parting</u> **Trennung** Scheiden, trennen séparation separación отделение spartimento пробирный камень / piedra de toque / touchstone / Probierstein / toetssteen / pierre de pietra di проба на touchstone test **Strichprobe** prueba de toque

touche, touchau

paragone

пробирном камне









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ESSENTIAL FIRE ASSAY TERMS

Oct-2024 Terms11_Hallm_Mic.doc

SAMPLE Preparation

FLUXING

FUSION

SLAG **CUPELLATION** Separation

PARTING

ANNEALING

AS USED IN BIBLIOGRAPHY

"HALLMARK (-ING)"

AUTHOR

CONTEXT

IAAO

Hallmarks are marks applied to precious metals to indicate the amount of pure metal in the alloy. Traditionally applied by striking with a punch, hallmarks can now also be applied using lasers.

IAAO

... hallmarking is carried out by assay offices which are situated in many countries over the world, but are most prevalent in Europe, the Middle-East and Asia. In some countries, assay offices are operated by the state and in others they are run as private operations. Some countries operate compulsory systems, others operate voluntary systems. The vast majority, but not all offices, are independent of the customers they serve. For a detailed summary of National gold jewellery caratages and marking requirements please click here (pdf 183 KBytes)

"MICRO-CUPELLATION"

AUTHOR

CONTEXT

Corti

Sample size is typically about 250 mg, although microcupellation techniques allow for much smaller samples of about 10 mg with consequent shorter processing time.

At such small sample sizes, ensuring that it is representative of the bulk alloy becomes more difficult.

upellation has become established in recent years, within the context of the cupellation of gold according to ISO 11426; with only about 10 mg of material taken from concealed places of a jewellery article by scraping, microcupellation can be carried out in a short time as clearly described in Table 11. (see Table 11 within full article: "Analysis of Carat Gold", GT, no 22, Jul. 1997).

Brill

...very accurate gold fineness (determinations) are obtained by microcupellation. Of course, a microbalance is essential for the weighing procedure; otherwise the usual equipment specified in ISO Standard 11426 can be employed.

MICRO-CUPELLATION AND ITS IMPLEMENTATION — METHOD OF THE IRISH ASSAY OFFICE:

Proposal ISO 11426 amended for small test portions

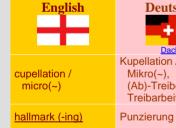
- Test portion 9 mg to 10.5 mg, weighed to ± 0.001 mg
- For samples containing approx. 999% gold, add 0.8 mg of copper to test portions of c.10 mg

 Lead foil 2 a **Brill**

- Furnace temperature approx. 1065° C
- Duration in furnace 8.5 to 9 minutes.
- Omit brushing precious metal buttons

As for ISO 11426 but with the following amendments:

- It is optional to anneal beads after flattening them on anvil
- It is optional to roll them but, if omitted, aim to achieve strip thickness of 0.12 mm to 0.15 mm by hammering and follow by annealing.
- Omit rolling strips into cornets if too small to do so
- Duration of samples in acid bath (density 1.2 g/cm³) in minutes
- Duration of samples in acid bath (density 1.3 g/cm³) in minutes
- Where Pt basket used, place the basket with the gold samples contained in silica thimbles for about 2 - 3 min. in a muffle furnace heated to ~900°C



Mikro(~) (Ab)-Treiben, Treibarbeit Punzierung Probierstein / touchstone test

Strichprobe

micro(~) waarmerk / keur rijksstempel

toetssteen / toetsstreek

cupellatie /

Français

Francophonie.org

micro(~) poinçonnage poinçon de l'État

coupellation /

pierre de touche, touchau Italiano

coppellazione / micro(~)

punzonatura /_ bollatura, bollo

pietra di paragone Castellano

copelación / micro(~)

punzonado contraste

piedra de toque / prueba de toque Русский

купелирование / микро(~) / купеляция капельный метод пуансон / пробирное

клеймо пробирный камень / проба на пробирном

Articles

Corti1

Corti2

▼

Corti3

Brill1

Evans

Wälchi1

Wälchi3

Young

Battaini

камне

FIRE ASSAY

touchstone /

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(free download)



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Dr. C. W. Corti \Diamond Assaying of Gold Jewellery - Review of Methods for Measuring Gold Content Dr. C. W. Corti

♦ Gold Analysis - Comparisons of available Techniques

○ 3.2 Articles from ►"Gold Technology" Magazine (WGC) ♦ "Assaying of Gold Jewel. - Choice of Technique" (GT, nº 32, 2001)

♦ "Analysis of Carat Gold" ♦ "Assaying and Hallmarking in London"

◊ "Analysis of Gold. A Review of Methods"

♦ "Fire Assay Reloaded"

♦ "Assaying Gold in Switzerland" ♦ "Touchstone Testing of Precious Metals"

(GT, nº 22, Jul. 1997) (GT, nº 3, Jan 1991) (GT, nº 3, Jan 1991)

D. W. Evans Wälchi / Vuilleumier Wälchi / Vuilleumier

Dr. C. W. Corti

M. Brill

(GT, nº 3, Jan 1991) 3.3 Articles from ►"Gold Bulletin" Journal (WGC) (GB, Vol. 13, issue 1)

Roland S. Young (GB, Vol. 47, issue 1) Battaini / Bemporad / Felicis

> **David Court** Court vLoon

♦ "The Art & Science of a Precious Metal Lab." (Alch., nº 48, LBMA) o 3.5 Articles from "Pure & Applied Chemistry" Magazine

3.4 Articles from ▶"Alchemist" Journal , (LBMA)

♦ "Analytical Chemistry of Noble Metals" (Pure & Appl. Chem., Vol. 49)

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WARE-

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ESSENTIAL FIRE ASSAY TERMS

Oct-2024 Terms12_Cupel_Kinds.doc

SAMPLE Preparation

FLUXING

FUSION

SLAG Separation

CUPELLATION

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MEANINGS & CLUES

CITATIONS FROM AUTHORITATIVE TEXTBOOKS & REFERENCE MANUALS

"CUPELS": ♦ MAGNESIA ♦ BONE ASH ♦ CEMENT — BEHAVIOUR

AUTHOR

CONTEXT

Bugbee

The cupel is a shallow, porous dish made of bone-ash, Portland cement, magnesia or other refractory and non-corrosive material. (p. 89) Cupels should not crack when heated in the muffle and should be so strong that they will not break when handled with the tongs. Good cupels give, a slight metallic ring when struck together after air-drying. It is best to heat cupels slowly in the muffle as this lessens the chance of their cracking.

A good cupel should be perfectly smooth on the inside, and of the right porosity. If it is too dense, the time of cupellation is prolonged and the temperature of cupellation has to be higher, thus increasing, the loss of silver. If the cupel is too porous it is said that there is danger of a greater loss, due to the ease with which small particles of alloy can pass into the cupel. The bowl of the cupel should be made to hold a weight of lead equal to the weight of the cupel.

The shape of the cupel seems to influence the loss of precious metals. A flat, shallow one exposes a greater surface to oxidation and allows of Bugbee faster cupellation; it also gives a greater surface of contact between alloy and cupel, and as far as losses are due to direct absorption of alloy, it will of course increase these.

The writer, using the same bone-ash and cupel machine, and changing only the shape of the cupel, has found shallow cupels to give a much higher loss of silver. In doing this work it was found harder to obtain crystals of litharge with the shallow cupel without freezing, and it was very evident that a higher cupellation temperature was required for the shallow cupel. The reason for this is that in the case of the shallow cupel the molten alloy is more directly exposed to the current of air passing through the muffle, and consequently a higher muffle temperature has to be maintained to prevent freezing. T. K. Rose* also prefers deep cupels on account of smaller losses. French found shallow cupels less satisfactory on account of sprouting. (p. 92 - 93)

Magnesia cupels are very hard, which is an advantage in that they do not suffer so much breakage in shipment. They are always factory-made and are decidedly more expensive than bone-ash cupels, which may be home-made. Certain brands of magnesia cupels give an apparently lower loss of silver in cupelling than can be obtained with bone-ash cupels but it is a question how much of this is real and how much due to an increase in the amount of impurities retained in the silver beads.

<mark>ia cupels</mark> behave quite differently from ordinary <mark>bone-ash</mark> cupels, and the assayer who is accustomed to <mark>bone-ash</mark> cupels will have to learn cupelling over again when he starts using those made of magnesite. This difference in behaviour is due mainly to the different thermal properties of the two materials. Both the specific heat and the conductivity of magnesite are decidedly greater than those of bone-ash, so that with cupels of both Bugbee kinds running side by side, the lead on the magnesia cupel is comparatively dull while that on bone-ash is very bright. This is due to the greater conductivity of magnesite, which allows a more rapid dispersion of the heat of oxidation of the lead, with the result that magnesia cupels require a higher muffle temperature than do bone-ash cupels. An especially high finishing temperature is required for magnesite cupels, to insure the elimination of the last 1 or 2% of lead. A bone-ash cupel will finish in a muffle, the temperature of which is sufficient to cause uncovering, but this is not true of the magnesia cupel, because in this case the heat of oxidation of the lead is diffused too rapidly and is not conserved to help out at the finish

Magnesia cupels absorb about two-thirds of their own weight of litharge, those of cement about three-fourths of their weight of litharge. (p. 116 -117)

CUPELLATION IN CUPELS OF DIFFERENT MATERIAL (pages 102-104)

Bone-ash cupel, mean specific heat between 15° and 100° C is 0.185. Magnesia cupel, mean specific heat for same temperatures is 0.215. A bone-ash and magnesia cupel of identical volumes weigh respectively 22 and 29 g. The heat conductivity of magnesia cupels is very much greater than that of bone-ash cupels. When the two types of cupels are heated to 90° C in a steam bath, at the end of 14 minutes the magnesia cupels are at 90° C and the bone-ash cupels at only 60° C. During cupellation of lead at the end of 6 minutes from the addition of the button the magnesia cupe showed practically the same temperature in the cupelling lead as in the bottom of the cupel, viz. 920° C, while the bone-ash cupel in the same muffle showed a temperature of 990° C for the cupelling lead, and only, 932° C in the bottom. The total heat capacity of a magnesia cupel is more than 50 %, greater than that of a bone-ash cupel of the same volume, so that on cooling the two types of cupel the magnesia cupel retains a higher temperature somewhat longer than the bone-ash cupel in spite of its greater diffusivity of heat. From this data the reason of the behaviour of magnesia and bone-ash cupels during cupellation is apparent. It will be noted:

- (1) That in magnesia cupels the lead is less bright and hence at a lower temperature than in bone-ash cupels, although the muffle temperature is Fulton the same. This is due to the fact that the extra heat generated by the combustion of the lead is diffused as rapidly as generated by the superior diffusivity of the magnesia cupel and hence cannot serve to raise the temperature of the lead, as is the case in the bone-ash cupel. Hence for the same "muffle temperature" the actual cupellation temperature of the lead in the magnesia cupels is 50° to 60° C lower than in the bone-ash cupels. To this fact is due the lower losses of precious metal in magnesia than in bone-ash cupels. From the discussion under "cupellation temperature" it will have been noted that with bone-ash cupels, if once the muffle has attained a temperature sufficiently high to cause the uncovering of the button, the rise in temperature of the lead due to its oxidation, is sufficient to carry the cupellation to a finish provided the muffle temperature is not lowered at the end of the operation. This is not the case with magnesia cupels for now obvious reasons and it will be necessary to raise the muffle temperature toward the end of the operation or what amounts to the same thing, push the cupel to the hotter part of the muffle. Assayers who are used to boneash cupels, therefore, have some difficulty at first due to "freezing" of buttons when using <mark>magnesia</mark> cupe
 - (2). Magnesia cupels retain a higher temperature longer than bone-ash cupels when withdrawn from the furnace or moved to the cool part of the muffle, and hence silver buttons show a lesser tendency to sprout, due to the slow cooling they undergo.

The lead in magnesia cupels seems to open somewhat more readily and cupels slightly faster than in bone-ash cupels. (p. 102 - 104)

- A cupel is a porous cylinder or inverted-cone frustum of refractory material with a cupped depression in the upper end for retaining the Shepard lead button. In modern practice, cupels are made of bone ash, cement, bone-ash-cement mixtures, or magnesia. Magnesia cupels are purchased as a finished product, but the others are usually made at the assay office. (p. 46 - 47)

Bone-ash cupels absorb a weight of litharge about equal to their own, cement cupels absorb slightly less than their weight, and magnesia cupels Shepard absorb three-fourths of their weight. Magnesia cupels are denser than bone-ash or cement cupels, hence a magnesia cupel of a given volume absorbs as much litharge as a bone-ash cupel of the same volume. (p. 47)

Magnesia cupels have a higher heat capacity and thermal conductivity than bone-ash or cement cupels, and hence the heat of oxidation of the Shepard dwelling lead is abstracted more rapidly. The alloy is therefore maintained at a lower temperature than with bone-ash or cement cupels, but higher muffle temperatures must be maintained throughout the cupellation cycle. Largely on account of lower alloy temperature near the finish of cupellation the loss of silver by cupel absorption is greatly decreased and is usually less than half of the loss obtained with bone-ash cupels, under analogous cupellation conditions. The gold loss with magnesia cupels is the same as with bone-ash cupels. (p. 51)

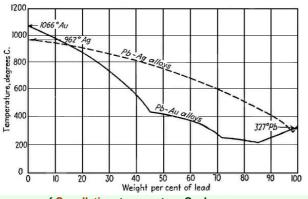
The heat of oxidation of the lead causes the temperature of the button to rise considerably above that of the cupel and the muffle, in the case of one-ash or <mark>cement cupels</mark>, but only slightly above the <mark>cupel</mark> temperature with <mark>magnesia cupels</mark>. Therefore with bone-ash or cement cupels the muffle temperature should be lowered during the driving period in order to keep the lead temperature from rising more than the minimum necessary for the reaction to proceed.

With magnesia cupels the temperature gradient from lead to cupel to furnace is not so great as with bone-ash or cement cupels, because of the Shepard greater heat capacity of magnesia compared with the other materials. The button will appear slightly hotter than the cupel, but the observable difference is by no means so great as with the bone-ash or cement cupels. Since the minimum lead temperature must be the same in all cases the

muffle temperature with magnesia cupels must be higher during the driving period than with other types.

Muffle temperatures of 870 to 880°C, as measured ½" above the muffle floor behind the cupel, are recommended for the driving stage of cupellation in magnesia cupels, and temperatures as low as 830°C may often be successfully used during the period of most active driving. (p. 58 -59)

Shepard



With magnesia cupels the temperature gradient from lead to cupel to furnace is not so great as with bone-ash or cement cupels, because of the greater heat capacity of magnesia compared with the other materials. The button will appear slightly hotter than the cupel, but the observable difference is by no means so great as with the bone-ash or cement cupels. Since the minimum lead temperature must be the same in all cases the muffle temperature with magnesia cupels must be higher during the driving period than with other types.

Muffle temperatures of 870 to 880°C, as measured $\frac{1}{2}$ " above the muffle floor behind the cupel, are recommended for the driving stage of cupellation in magnesia cupels, and temperatures as low as 830°C may often be successfully used during the period of most active driving. (p. 58 -

■ MELTING POINTS OF **LEAD-SILVER** & **LEAD-GOLD** ALLOYS

Summary of Cupellation-temperature Cycle.

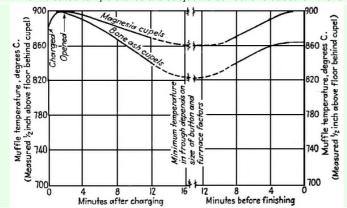
Characteristic temperature cycles in cupellation are shown on Fig. 8, based on measurements taken with a pyrometer ½" above the muffle floor iust behind the front row of cupels. The curves apply particularly to buttons from 20 to 25 a in weight and with beads weighing 50 ma or more.

cupelled with feathers.

Shepard

Smith

The indicated temperatures are subject to corrections based on the furnace draft, heating and cooling lag, and the pyrometer position.



The minimum temperature allowable during the driving depends also upon the type of cupel used and the size of the button and bead. Large buttons that are cupelled rapidly in bone-ash cupels with the ordinary shallow cup frequently permit minimum muffle temperatures as low as 790°C and will finish at 820°C if the beads are small.

Magnesia cupels under similar conditions require minima of 830°C in the driving trough, and 840° to 850°C to finish.

The formation of feathers of litharge can be observed readily with boneash or bone-ash-cement cupels and serves to indicate proper driving temperature, but when copious feathers form on magnesia cupels the temperature is dangerously near the freezing point.

Finishing temperatures greatly in excess of 900°C, as measured in the manner indicated above, should be avoided in all cases, as higher temperatures cause greatly increased losses of gold and silver with all types of cupels and all variations in button and bead weight. (p. 66 - 67) **◆**CHARACTERISTIC CUPELLATION-TEMPERATURE CYCLES

For example, the temperature of the cupeling alloy in a dense magnesia cupel does not rise so far above muffle temperature as it does in a bone-Shepard ash cupel, because the magnesia cupel is a better conductor of heat. This is the principal reason for good magnesia cupels giving lower losses than one-ash cupels. (p. 231)

– <mark>Magnesia</mark> was introduced a few years ago, as a substitute for <mark>bone-ash</mark> and a large number of brands of so-called "patent" Magnesia Cupels cupels made with a magnesia base are now on the market. The <mark>magnesia</mark> (MgO) is produced by strongly calcining crude mineral <mark>magnesit</mark> (magnesium carbonate, MgO,CO₂). The cupels require to be made under high pressure and should be baked at a high temperature before use. They are then very hard and firm and of a brown colour, resembling fireclay. Owing to the high pressure required (usually hydraulic), magnesia cupel cannot satisfactorily be made in the laboratory like bone-ash cupels. (p. 49)

Cupellation on Cupels of Material other than Bone-ash — Since, as stated on page 49, a large number of cupels made of materials other than one-ash (mainly magnesia) are now supplied for assay purposes, it is well to point out that during cupellation there is a considerable difference in the behaviour of these so-called "patent" cupels as compared with bone-ash cupels, due to differences in the thermal properties of the materials used.1

The diffusivity of heat and the specific heat of magnesia cupels are greater than those of bone-ash cupels. If similar cupellations be conducted in p<mark>one-ash</mark> and in <mark>magnesia cupels</mark> side by side, a marked difference will be seen in the behaviour of the lead. The lead on the bone-ash cupel during the cupellation is very bright, whereas the lead on the magnesia cupel is comparatively dull during a considerable part of the operation and is not so hot, although the muffle temperature is the same for both. "This is due to the fact that the extra heat generated by the oxidation of the lead is diffused as soon as it is generated, owing to the superior diffusivity of the magnesia cupel and hence cannot serve to raise the temperature of the lead, as is the case in the bone-ash cupel. Hence for the same 'muffle temperature' the actual cupellation temperature of the lead in the magnesia cupels is 50° to 60° C lower than in the bone-ash cupels

It has been pointed out on page 164 that the heat generated by the oxidation of the lead is sufficient to carry the cupellation to a finish with boneash cupels, provided the muffle temperature is not lowered at the end of the operation; but, for the reasons stated above, it is necessary in the case of magnesia cupels to employ a slightly higher temperature during the cupellation and to raise the temperature towards the end of the operation.

The difference between the temperatures of the lead during cupellation on <mark>magnesia</mark> and on <mark>bone-ash cupels</mark>, which can be noticed by observation, is much more marked at the beginning of the cupellation, and, in fact, is hardly discernible at the very end of the operation.

It will be noted also that magnesia cupels retain heat longer than bone-ash cupels, consequently silver beads take longer to solidify and to spit on magnesia than on bone-ash cupels, after being withdrawn from the same muffle temperature. Silver beads are also much less liable to spit on <mark>nagnesia</mark> than on <mark>bone-ash</mark> cupels and the nature of the spit is different in the two cases, the spit in the former case generally taking the form of a frosty appearance only instead of the well-known "vegetation" obtained with bone-ash. These important differences in the properties of magnesia and bone-ash cupels are not always recognised by assayers.

An assayer, when asked to test magnesia cupels, usually puts half a dozen in the muffle with half a dozen bone-ash and cupels under conditions suitable for the bone-ash, with the result that he forms an unfavourable opinion of the magnesia cupels. When using magnesia cupels under these conditions the lead is very liable to "freeze" and the results are unsatisfactory; but if the proper conditions for magnesia cupels are employed, the results are quite as satisfactory as those obtained with bone-ash. Although a somewhat higher temperature is required for cupellation on magnesia cupels, the tendency is to have a very much higher temperature than is necessary, in order to make the cupelling lead look like that on bone-ash cupels; and this is a great mistake, whereby one of the most important advantages of the magnesia cupels is lost. The loss of silver due to absorption is usually less with magnesia than with bone-ash cupels (see p. 177).

Portland Cement Cupels — The behaviour of Portland cement cupels during cupellation is very similar to that of bone-ash cupels, although, as

shown on page 177, the loss by absorption is slightly higher. When using cupels made entirely of Portland cement for gold assays, especial care must be taken to thoroughly clean the buttons, otherwise when subsequently parting in nitric acid insoluble silica will remain adhering to the cornet and be weighed as gold. This difficulty may be overcome by "facing" the cement cupel with bone-ash. In this case the cupel mould is filled about two-thirds full with cement and bone-ash added to fill the mould, the cupel then being finished .in the ordinary way.

ement cupels are also made from mixtures of Portland cement and bone-ash, the proportions being usually equal parts of each. (pages 166-167) At present there is little evidence as to the comparative variations in the absorption of silver and gold by bone-ash and magnesia cupels

Smith respectively, but it would appear from the data published that the absorption is less with magnesia cupels than with bone-ash. Whatever cupels are used, the absorption should be tested frequently. (p. 176) With ordinary gold-bullion assays the absorption of gold is stated by S. Smith 1 to be about 0.5 parts in 1000 for bone-ash cupels and about 0.3

Smith parts in 1000 in the case of Morganite cupels. It was found that this absorption difference for cupels of these materials was practically constant. (p. Cupellation is an oxidizing fusion in a vessel that repels molten metal and absorbs metal oxides. Metal oxides wet and absorb into the vessel

Because precious metals, such as gold, silver, and platinum group metals, resist oxidation, cupellation ends with all precious metals concentrated in the form of a doré bead

Gilbert

Bone ash (calcium phosphate): Made from calcined sheep bones. Its low thermal conductivity allows lower furnace cupellation temperatures. The oxidation of lead generates heat. Temperature, at the lead surface, increases over the general furnace temperature.

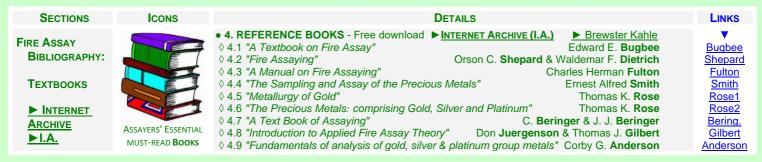
Cement (lime & clay): Raw material is very cheap. Cement loses water of combination when heating and then physical imperfections are produced on cup's surface. This can trap the doré. Bone ash / Cement: Composition is cheap. Physical imperfections, which form as the cement dehydrates, trap the doré.

Magnesia (magnesium oxide): Give more accurate silver assay values. More tolerant of the effects of cupelling dirty (slag dust retained) buttons.

(cupel) wall until only oxidation-resistant metals (i.e., precious metals on the bottom of the electromotive scale) remain.

The type more commonly used in F.A. labs (p. 54)

English	Deutsch +	Nederlans 111	Français O	Italiano	Castellano	Русский
	<u>Dach</u>	<u>Taalunie.org</u>	Francophonie.org	Accademiadellacrusca.it		Ruslang.ru
bone ash	Knochenasche	bot as	cendre d'os	cenere d'ossa	ceniza de hueso	костяная мука
cupel / cupels	Kupelle / (~)n	cupel	coupelle /coupelles	coppella / coppelle	copela / copelas	капель / капели
cupel (to)	kupellieren / treiben/ ab(~)	cupelleren / afdrijven	Coupeller	coppellare	copelar	Купелировать
cupellation / micro(~)	Kupellation / Mikro(~), (Ab)-Treiben, Treibarbeit	cupellatie / micro(~)	coupellation / micro(~)	coppellazione / micro(~)	copelación / micro(~)	купелирование / микро(~) / купеляция капельный метод
cement	Zement	cement	Ciment	cemento	cemento	цемент
magnesia	Magnesia	magnesia	magnésie	magnesia	magnesia	окись магния, магнезия









FIRE ASSAY SL - CARRER DE NOVELL, 54 - 2nd FLOOR ES-08014 BARCELONA - SPAIN - EU

WARE-

TSM Log. – B-682, CTRA. ACCÉS COSTA BRAVA km 1,4 WARE-HOUSE TSM LOG. — B-682, CTRA. ACCÉS COSTA BI ES-08389 PALAFOLLS — BCN — SPAIN — EU



ESSENTIAL FIRE ASSAY TERMS

Oct-2024 Terms13_Scorification.doc

SAMPLE Preparation

FLUXING

FUSION

SLAG Separation

CUPELLATION

PARTING

ANNEALING

MEANINGS & CLUES

CITATIONS FROM AUTHORITATIVE TEXTBOOKS & REFERENCE MANUALS

"SCORIFICATION": SCORIFIERS

AUTHOR

CONTEXT

corifiers are shallow fire-clay dishes used in the scorification assay of gold and silver ores. They should be smooth on the inside, dense and Bugbee impermeable to lead and slag and should be composed so as to withstand, as much as possible, the corrosive action of litharge. (p. 33)

fication assay is the simplest method for the determination of gold and silver in ores and furnace products. It consists simply of an oxidizing muffle fusion of the ore with granulated lead and borax-glass.

The lead oxide formed combines with the silica of the ore and also to a certain extent dissolves the oxides of the other metals. The only reagents

used other than lead are borax-glass and occasionally powdered silica, which aid in the slagging of the basic oxides.

The scorifier is a shallow, circular fire-clay dish 2" or 3" in diameter. The sizes most commonly used are 2½", 2¾" and 3" in diameter.

The amount of ore used varies from 0.05 A. T. to 0.25 A. T., the amount most commonly used being 0.10 A. T.

having an acid gangue, it is generally satisfactory and widely used.

With this is used from 30 g to 70 g of test lead and from 1 g to 5 g of borax-glass, depending on the amount of base metal impurities present. Sometimes powdered silica and occasionally litharge are also used.

Bugbee With nearly pure galena, or a mixture of galena & silica, a charge of 30 g to 35 g of test lead and 1 g of borax-glass will suffice for 0.10 A. T., of ore, but when the ore contains nickel, copper, cobalt, arsenic, antimony, zinc, iron, tin, etc., a larger and larger amount of lead and borax-glass must

be used according to the relative ease with which the metals are oxidized and the solubility of their oxides in the slags formed. Of the above, copper especially is very difficultly oxidized and when much is present in the ore the lead button from the first scorification will have corified once or twice with added lead.

Iron, on the other hand, is comparatively readily oxidized, and except for the necessity of adding an extra amount of lead and borax-glass to

make a fluid slag the ore is as readily assayed as galena. Lime, zinc, and antimony require especially large amounts of borax-glass to convert their refractory oxides into a fusible slag. (p. 127) The scorification assay is simple, inexpensive and reasonably rapid. For the determination of silver in sulphide ores

Bugbee

Fulton



It is particularly suited for the determination of silver in ores containing considerable amounts of the sulphides, arsenides or antimonides of the difficultly oxidizable base metals, particularly copper, nickel and cobalt. It is used in

many localities for silver in all sulphide ores, as well as for gold and silver in copper bullion, impure lead bullion, copper and nickel Mattes and Speiss.(p. 134 - 135) SCORIFICATION. - This is the oxidizing fusion of ore with metallic lead in the

900 386 850 800 764 750 725 -732° 700 (Not 650 P60-573, 600L Composition, weight per cent SiO₂

muffle-furnace, producing, in the main, a litharge slag, i.e., an oxide slag. It is a method of assay which requires no previous preparation of the ore or preliminary assay, and as practically only one flux is employed, it is both a cheap and a rapid method.

It is also a thoroughly reliable method, when proper precautions are taken and when it is employed on material suitable for the purpose. The operation is performed in shallow fire-clay dishes, called scorifiers.

Before these dishes are used it is usual to line the inside with ferric oxide

This is done by preparing crushed iron ore or ochre, mixing with water, and painting the inside of the dishes.

This gives them a basic lining, and to some extent prevents the oxide slag from attacking the silica in the clay.(p. 122) (*)

(*) Eutectics of PbO-SiO₂ system with **low melting points**: 710° – 716° – 732°C ■ Reason why Litharge causes wall perforation

on silica refractory clays of scorifiers

MELTING POINTS DIAGRAM - SYSTEM PbO-SiO₂ ion may be divided into the following distinct steps:

1. Melting. In this stage the lead melts, and the ore, being of a lesser gravity, rises to the surface of the molten lead and floats there.

2. Roasting. The ore on the surface of the lead is attacked by the oxygen of the air and roasts in the same way as described under "Roasting of Ores.

v<mark>rification</mark> Proper. The l<mark>ead</mark> commences to oxidize, forming <mark>litharge</mark>. A small percentage volatilizes and the balance forms a fusible slag. This now absorbs the oxides formed by the roasting, dissolving them and forming an igneous solution.

The silver and gold, liberated, are absorbed by the remaining metallic lead. **Fulton**

The slag, as it forms, drops to the side, forming a slag ring, with the center of the lead bath open to the atmosphere. The reason for this is that the meniscus of molten lead is convex, thus causing the collecting of the slag on the rim of the scorifier

<mark>ation</mark> continues until the whole of the <mark>lead</mark> is covered over with slag. It is then considered finished and the assay is poured.

Should the assay be left in the muffle, the lead will still continue to oxidize, although none is exposed to the air, the interchange of oxygen taking place by means of the litharge and other oxides present.

The size of the lead button desired from this assay ranges from 15 to 20 g. If the scorification is continued to produce smaller buttons, losses are apt to occur by oxidation of the silver, especially if this is present in considerable amounts, thus forming rich slags. (p. 123)

The scorification assay is an oxidizing fusion in a shallow fire-clay dish, known as a "scorifier," of a relatively small quantity of ore and a minimum

of acid fluxes with a comparatively large amount of granulated lead. Oxidizing conditions are maintained by admitting air to the muffle, which oxidizes a part of the lead and roasts the ore. Shepard The fused litharge combines with silica, borax glass, and the oxidized base metals of the ore to form a slag.

Precious metals are collected by the molten lead and remain alloyed with the lead button. The buttons from the scorification are cupelled, weighed, and parted as in the crucible assay. (p. 165)

APPLICATIONS AND LIMITATIONS. The scorification method is best adapted to the assay of ores (1) that are sufficiently rich in gold or silver so that weighable beads are obtained with ore samples not exceeding 0.2 assay ton,

Shepard and usually 0.1 assay ton, in weight; (2) that are so homogeneous that a small sample is sufficiently reliable;

(4) that contain oxidizable impurities but not any appreciable quantity of decrepitating compounds. (p. 165) One of the useful applications of scorification is the retreatment of crucible assay buttons, either to remove certain impurities, to decrease their size, or to combine two or more crucible buttons in case the crucible charge is so large as to require distribution between two or more crucibles.

(3) that are relatively free from basic oxides and from metals, other than mercury, below lead in the electromotive force series of elements; &

The principal advantages of scorification over crucible assaying are that the control of button size is independent of the nature of the ore, the cost of fluxes is low, less time is required to prepare the charges for melting, and the muffle capacity for scorifiers can be made greater by stacking a second layer of sc rs on the rims of the first laver.

Shepard These advantages are largely offset by the narrow limitations of the method with respect to the size and character of the sample, and by a longer heating cycle in the furnace. The cost of fire-clay ware is nearly equal for the two processes, because scorifying dishes, though cheaper, are ordinarily usable only once,

whereas the average life of crucibles is 3 to 4 fusions. On ores of types suited to ideal scorification, the accuracy of scorification compares favorably with the crucible (p. 166) orification. This operation consists of an oxidising fusion of an ore with metallic lead in the muffle furnace and has for its object

Smith



the concentration of the precious metals in a button of metallic lead and the removal of every other constituent of the ore by the solvent action of molten litharge formed during the operation by the oxidation of part of the lead added. <mark>orification</mark> is conducted in a <mark>scorifier</mark>, which must be well dried at a gentle heat for ten or fifteen minutes before use. If this precaution is not taken, "spurting" of the lead may occur during the operation. (p. 155)

Smith



corrosive action of the molten litharge. See note (*) and M P diagram at "Fulton" above

Sometimes the scorifier is coated inside with ferric oxide to protect it as far as possible from the

A thin paste made by mixing powdered haematite with water is used for the purpose.

The amount of ore and lead used varies according to the nature of the ore, being on the average about ten to twenty parts of lead to one part of ore. (p. 156) corification may sometimes be used as a substitute for fusion assay. It is used extensively for certain types of silver and gold assays such as

bullion or recycled high-grade precious metals. In this instances it involves mixing the ore sample with lead, and covering with borax. The reactions involved are similar to the pot fusion, but there is also a series of reactions between air and the constituents of the charge. The

oxidised lead forms part of the fusion mixture, and the residual lead acts as a collector.

The scorification assay is not, in general, recommended for accurate noble metal determination in minerals. **Anderson** The process is essential, however, for the reduction of button size or for cleaning a badly contaminated button. In this instance the button is

STEP 2

transferred to a scorifier, and covered with a few grams of borax and silica. At the required temperature of 1050°-1100°C and in the presence of air the melted lead is oxidised, and together with the borax and silica forms a slag with the oxidised base metals contaminants. The slag moves progressively to the periphery of the scorifier, and the molten lead forms the centre of eye of the fusion. The melt may then be

poured as in the crucible fusion.

Anderson See the 4 steps graphically illustrated in below:

SCORIFICATION STEPS EXPLAINED

<u>Anderson</u>





Molten

Lead





STEP 3

EYE OPEN

(DOOR OPEN)



Castellano

Eye Slag

Ring



STEP 4

EYE CLOSED

(DOOR CLOSED)

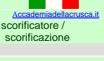
scorification



ICONS







DETAILS

escorificación

Brewster Kahle

Edward E. Bugbee

Ernest Alfred Smith

плавка

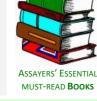
SECTIONS

FIRE ASSAY

ARCHIVE

<u>▶1.A.</u>

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LINKS