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Brass Particles on the Turin Shroud: Optical Microscopy Studies and SEM-EDX Analyses

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Abstract: We have explored by optical microscopy and scanning electron microscopy coupled with energy-dispersive X-ray twenty-two particles and formations of brass located in a sample of the Face area of the Turin Shroud. They differ by the proportions of copper and of zinc in the alloys. The nineteen brass particles are micro-sheets or micro-balls, generally of yellow colour. The p5 formation is representative of the cementation process of brass production, and the k15 formation is representative of the direct process. These brasses were loaded on the Turin Shroud surface at different times during history, to imitate gold powder.

Keywords: Turin Shroud, Face area, Brass, Optical microscopy, Scanning Electron Microscopy, Energy Dispersive X-ray

1. Introduction

The Turin Shroud (TS) is a well known linen tissue on which a body image is imprinted [**1**]. We have obtained a small triangular sticky tape that was sampled on its surface (corresponding to a part of the Face of this body) and we concentrated in the past years on the study of microscopic particles located on the surface of this sticky tape [**2**].

Brass is an alloy of copper (Cu) and zinc (Zn), in proportions which can be varied to achieve different colours. Brass will corrode with time ; this often happens when copper reacts with sulphur (S), to form a brown / black surface layer of copper sulphide.

In the present study we described in details, by optic microscopy and SEM-EDX analyses, all the brass particles (and formations containing its) that we have found on the triangle surface.

2. Material and Methods

The material is the small (1.36 mm height, 614 µm wide) sticky tape triangle at the surface of which all particles were deposited. For practical reasons, the surface of this triangle was subdivided into nineteen sub-samples areas, named A to S ; because of its complexity, the E area was subdivided into seven (a to g) sub-areas.

All the brass particles were first observed (to determinate colours) by optical microscopy (both in direct and inverted positions of the triangle) using a photo-microscope Zeiss (model III, 1972).

These particles were also observed, with any preparation, on the adherent part of the surface of the triangle. These observations were conducted by SEM (Scanning Electron Microscopy), using a Philips XL instrument (of the environmental version). GSE (Gazeous Secondary Electrons) and BSE (Back Scattered Electrons) procedures were used , the second one to better detect heavy elements. Elemental analysis for each particle observed were realized by EDX (Energy Dispersive X-ray), this SEM microscope being equipped with a Bruker probe AXS-EDX (the system analysis is PGT : Spirit Model of Princeton Gamma Technology).

The p5 formation was examined with another SEM apparatus (SEM2) : a FEI model Quanta 25 of FEG, both in LFD (Large Field Detector) and in CBS (Circular Back Scattering) procedures.

Each elemental analysis is given in the form of a spectrum, with kiloelectrons/Volts (Ke/V) on the abscissa and elemental peak heights in ordinates. Highly Resolutive (HR) spectras are those where the ordinate graduations are enhanced, to better see little element peaks.

Peak heights of the Kα1 rays of Cu and Zn elements were used to estimate qualitatively the Cu/Zn proportions in the brass particles ; peak heights of the L α1 rays of Cu and Zn were used to determinate these proportions, in a semi-quantitative fashion. Normal compositions are those where percentages of the Cu and Zn elements only were compared.

3. Results

We have found on the surface of the triangle a total number of nineteen (c1, c7, c33, e43, l in the E.c area, e130, f9, k1, k13, k16, k17, k37, I in the L area, m62, m63, m64, r15, r22 and s18) brass particles and three (b53, k15 and p15) formations containing it.

There is no brass particle in the area A of the triangle. The b53 formation, in the B area, contains brass. The SEM photograph of **Figure 1** shows the particles

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located on some part of the B area and, among them, the b53 formation. Adjacent particles to b53 in that area part are : b51, a losangic Diatom ; b50, a set of about ten micro-particles of calcium carbonate corresponding to broken Coccolith ; b52, a ceramic ; b49, a quartz ; b44, a calcium carbonate ; b45, a Diatom, b46, a silica ; b54-55, two pollens [**3**] ; b56, a calcite ; b57, a spore ; b58, a gypsum ; b59 an aragonite.

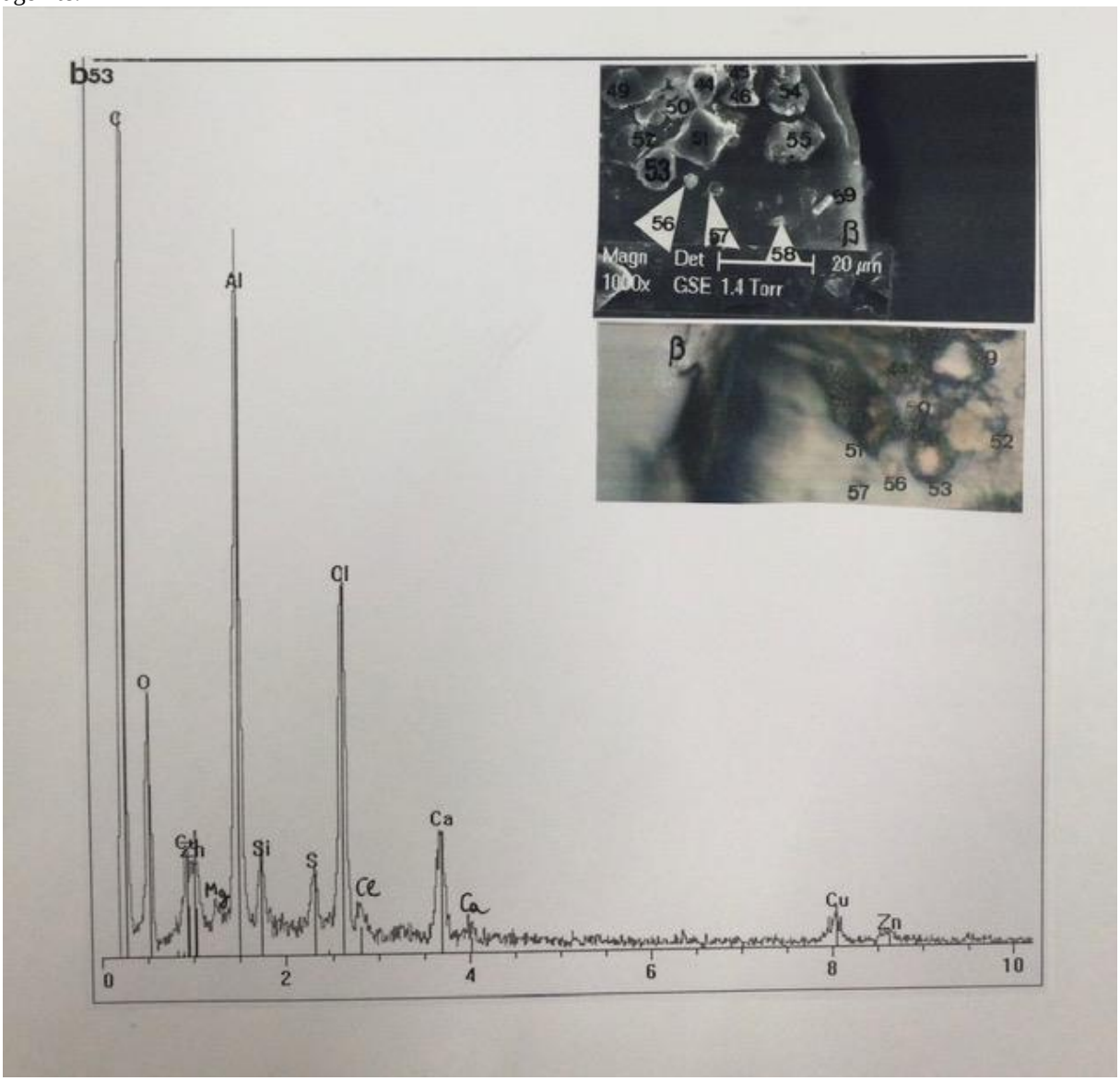


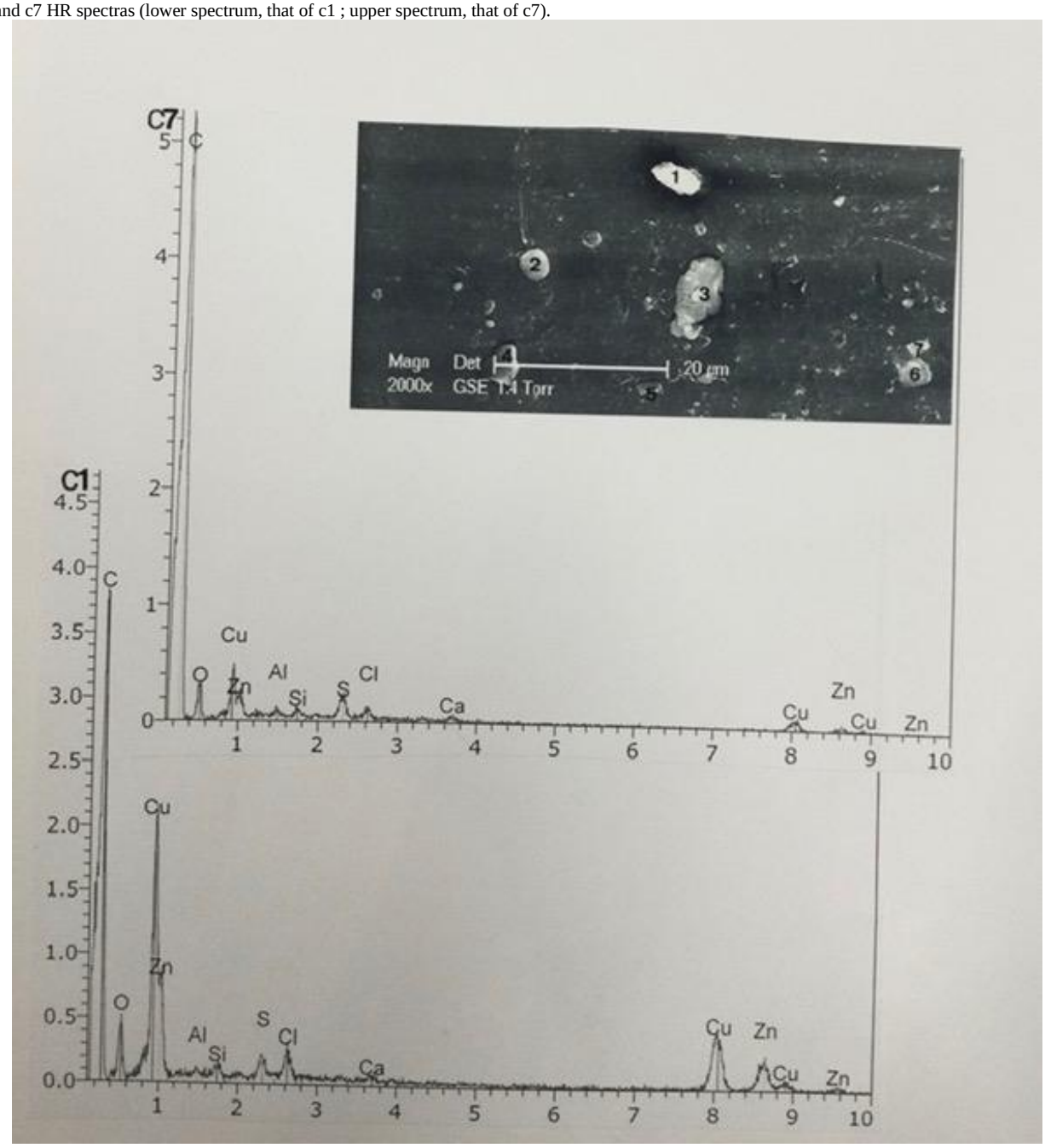
Figure 1. The b53 formation. *Upper photograph*. SEM photograph (1000x) in GSE, of some part of the side of the B area showing b53 (β : right border of the triangle). *Lower photograph*. Photograph in optic microscopy (1 200x), in inverted position, of this area part. *Below*. The b 53 spectrum. C : carbon ; O : oxygen ; Cu (two peaks) : copper ; Zn (two peaks) ; Mg : magnesium ; Al : aluminium ; Si : silicium ; S : sulphur ; Cl (two peaks) : chlorine ; Ca (two peaks) : calcium.

The b53 formation is an oval sheet, with a maximal dimension of 13.5 µm. Its spectrum is very rich in aluminium (Al) and has a relatively great peak of chlorine (Cl). In that spectrum, brass is constituted of equal proportions of Cu and Zn (Cu=Zn type).

In optic microscopy the b53 formation is of an intensive yellow colour.

We found three brass particles in the C area : c1, c7 and c33. The SEM photograph of **Figure 2** shows some part of the C area containing c1 and c7 particles. Neighbouring particles in that area are : c2, a rounded calcite ; c3, a Cyanophycae ; c4, a silica ; C5, a steatite ; C6, a little phosphorite with iron.

Figure 2. The c1 and c7 particles. *Above*. SEM photograph (2 000x), in GSE, of the upper part of the C area showing c1 and c7. *Below*. The c1 and c7 HR spectras (lower spectrum, that of c1 ; upper spectrum, that of c7).

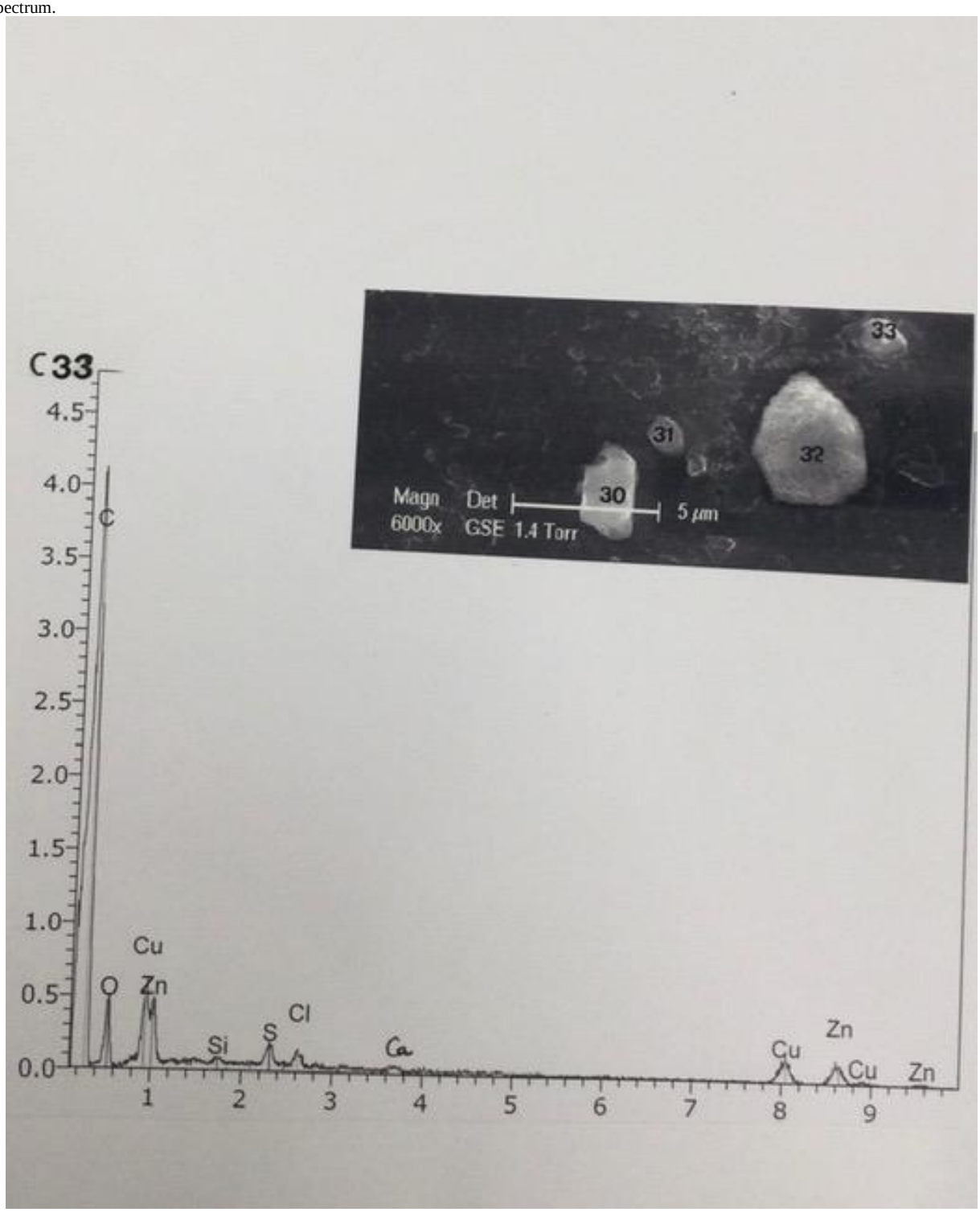


The c1 particle is elongated in form, (a micro-sheet), with a maximal length of 7.2 µm. In its spectrum, typical of brasses, the Cu proportion is approximatively double than that of Zn ; in our nomenclature, these proportions are summarized as Cu : 2 / Zn : 1. In optic microscopy the c1 colour is yellow-red.

The c7 particle is a micro-sheet of about 3.5 µm of length. In its spectrum the brass is of the Cu : 2/Zn : 1 type. Its colour is black in optic microscopy.

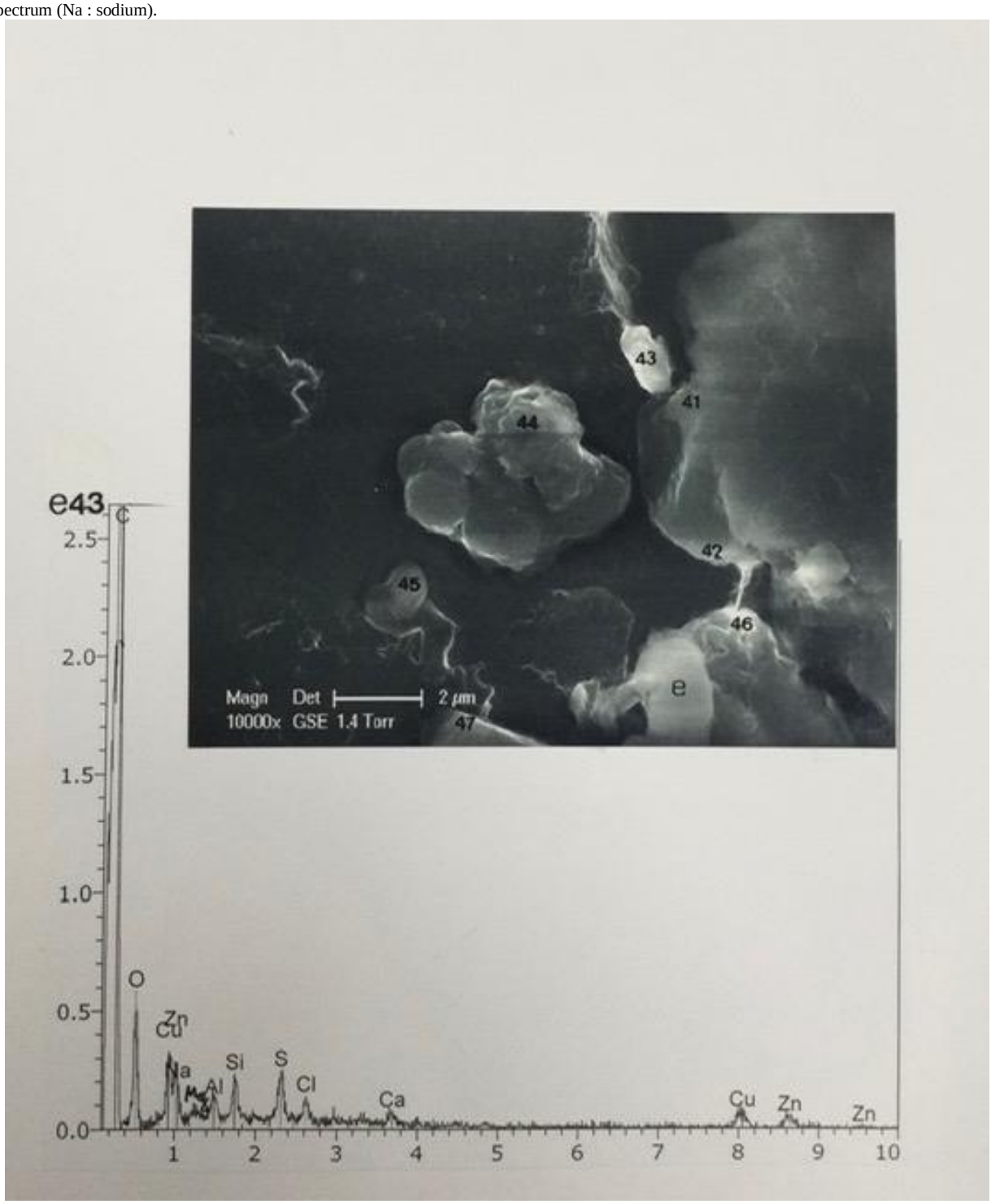
The SEM photograph of **Figure 3** shows some other part of the C area containing C33. Other particles in that area are : c32, a rare earth ; c31, an Ostracod ; c30, a calcium carbonate. The c33 particle is a micro-ball, of dimension of about 2.2µm. Its spectrum is of the Cu=Zn type. Its colour is yellow in optic microscopy.

Figure 3. The c33 particle. *Above*. SEM photograph (6 000x), in GSE, of the middle part of the C area showing c33. *Below*. The c33 HR spectrum.



There is not brass particle in the D area. We found three brass particles in the E area :e43, l (on e57) and e130. SEM photograph of **Figure 4** shows some part of the E.c area containing e43. Other neigbouring particles in that area are : e41, an alumina-silicate of potassium ; e42, a lapis-lazuli [**4**] ; e44, a clutch of Insect ; e45, an egg isolated from this clutch ; e46, a Cyanophycae ; e47, a glass fragment ; the e part of the SEM photograph is the upper extremity of the fourth linen fiber [**5**].

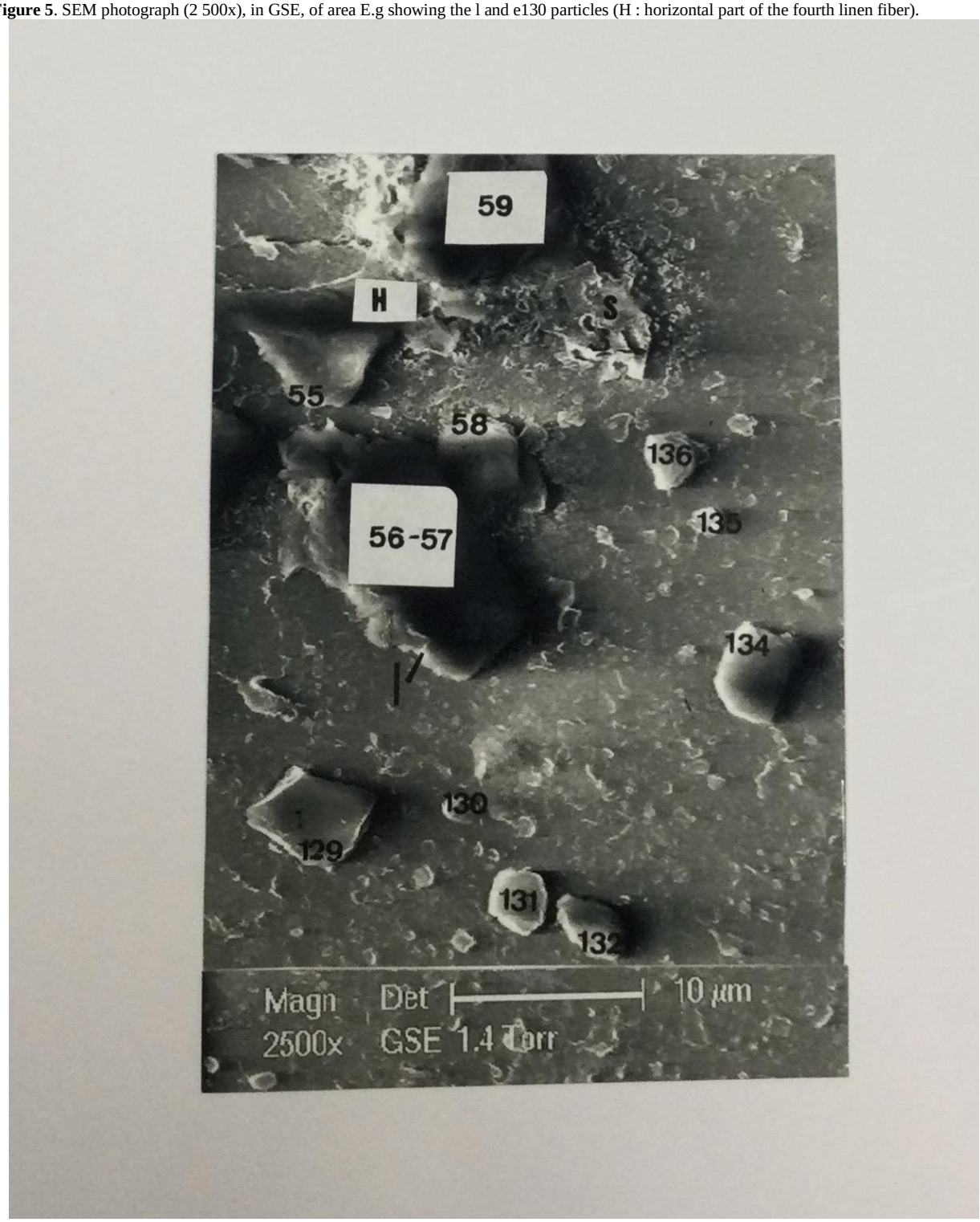
Figure 4. The e43 particle. *Above* : SEM photograph (10 000x), in GSE, of an upper part of the E.c area, showing e43. *Below*. The e43 HR spectrum (Na : sodium).



The e43 particle is a micro-sheet, of dimension of about 2.1 µm. In its spectrum, the Cu proportion is lightly superior to that of the Zn (the Cu > Zn type). Its colour is yellow in optic microscopy.

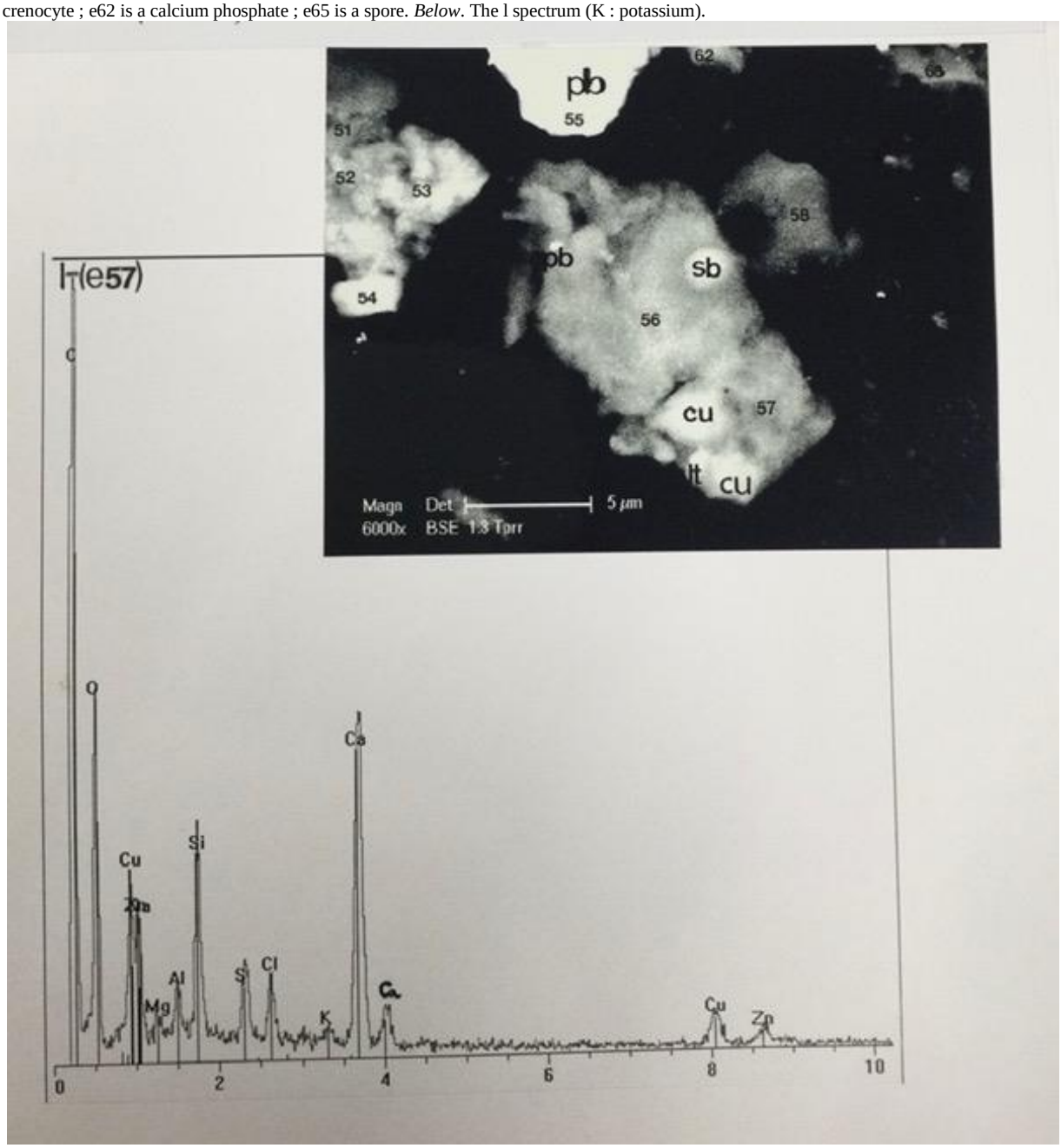
The SEM photograph of **Figure 5** shows some part of the E.g area containing the l particle (located on e57) and the e130 particle. Neighbouring particles in that area are : e55, a lead ; e56-57, two adjacent fragments of colourings ; e58, a crenocyte [**6**] ; e59, an osseous fragment [**7**] ; e129, a silica ; e131, an alumino-silicate ; e132, a double carbonate ; e134 and e135, two calcites ; e136, a gypsum.

Figure 5. SEM photograph (2 500x), in GSE, of area E.g showing the l and e130 particles (H : horizontal part of the fourth linen fiber).



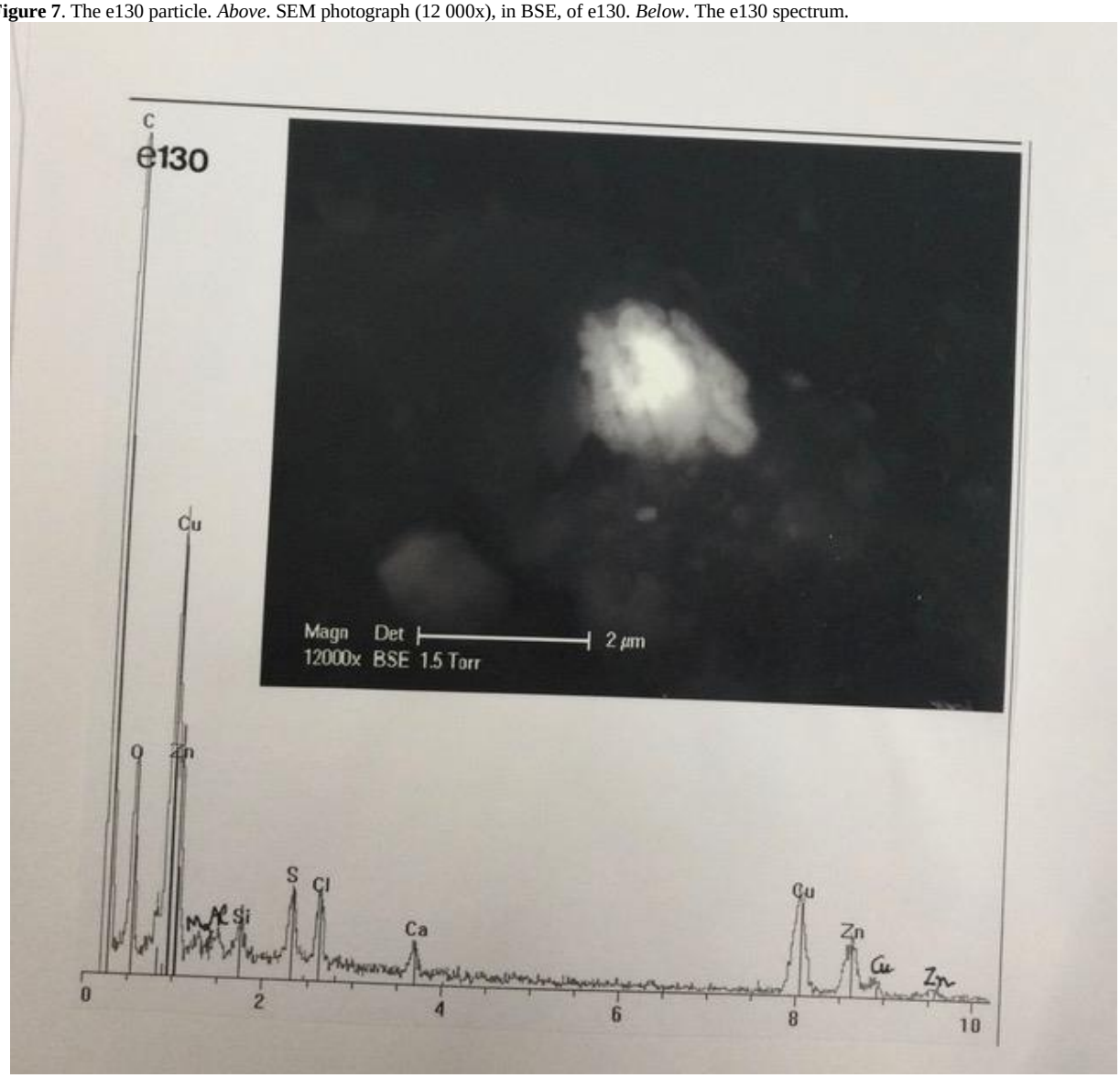
The SEM photograph of **Figure 6** shows some part of the E.g area containing the l particle located on e57. This l particle is a little micro-sheet of an approximate length of 0.9 µm. Because of its little size, corresponding to an "ultra-fine" particle, the l spectrum includes elements of the e57 substratum like silicium (Si) and calcium (Ca). The Cu proportion is higher than that of Zn (the Cu>>Zn type). Its colour is black in optic microscopy.

Figure 6. The l particle. *Above*. SEM photograph (6 000x), in BSE, of some part of the E.c and the E.g area showing the l particle (lt) on e57. The e57 particle (a painting fragment) has also two copper (Cu) sheets on its surface ; the e56 particle, another painting fragment , has lead (Pb) and barium sulphate (sb) sheets on its surface ; e51 is a PVC plastic ; e52 and e53 are lapis lazuli ; e54 is a marble fragment ; e55 is a lead ; e58 is a crenocyte ; e62 is a calcium phosphate ; e65 is a spore. *Below*. The l spectrum (K : potassium).



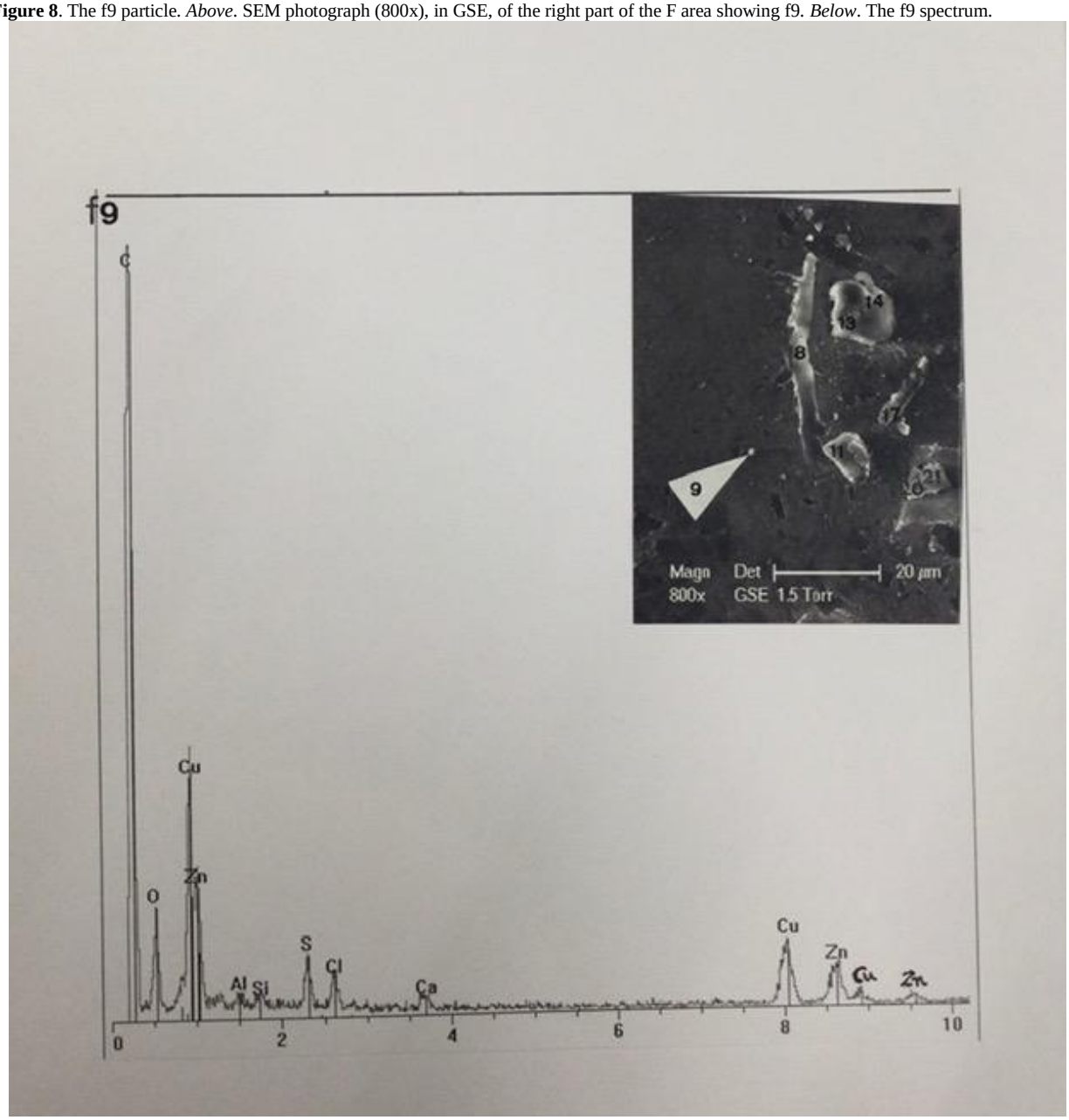
The SEM photograph of **Figure 7** shows the e130 particle (in some part of the E.g area). It is a very deteriorated micro-ball particle of about 2.3 µm of diameter. Its spectrum is of the Cu>>Zn type, more precise semi-quantitative measures showing the proportions of 70% of Cu and 30% of Zn. Its colour is yellow in optic microscopy.

Figure 7. The e130 particle. *Above*. SEM photograph (12 000x), in BSE, of e130. *Below*. The e130 spectrum.



The f9 particle is the only brass particle found in the F area. SEM photograph of **Figure 8** shows some part of the F area containing f9. Other neighbouring particles in that area are : f8, three cylindric piled up Diatoms ; f11, a triangular Diatom ; f13-14, a colouring fragment ; f17, a cylindrical Diatom ; f20 and f21, two triangular Diatoms.

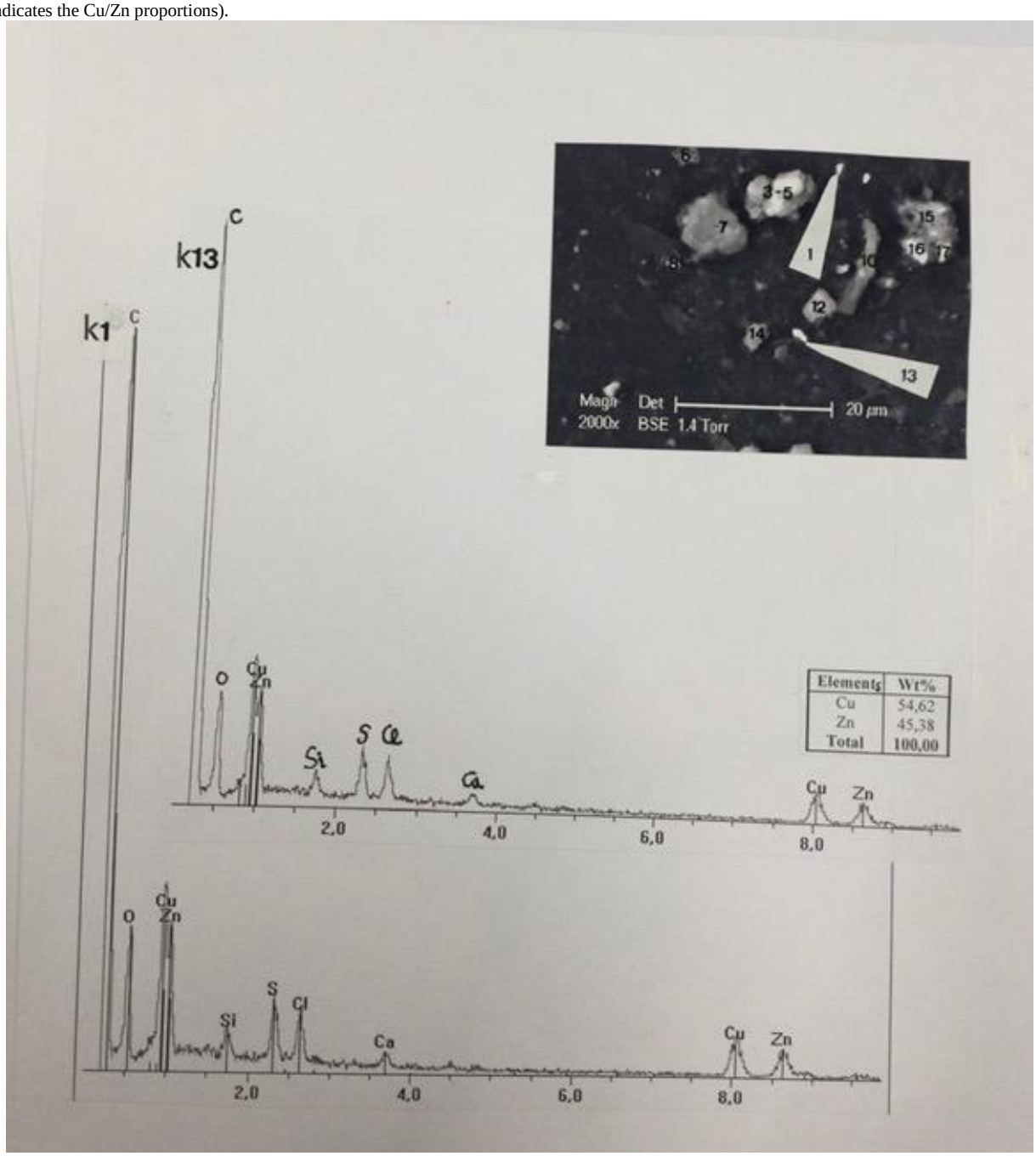
Figure 8. The f9 particle. *Above*. SEM photograph (800x), in GSE, of the right part of the F area showing f9. *Below*. The f9 spectrum.



The f9 particle is a micro-ball, of about 1.9 µm of diameter, of the Cu : 2/Zn : 1 type. Its colour is yellow in optic microscopy.

There is not brass particles in areas G,H, I and J. There are five (k1, k13, k16, k17 and k37) brass particles and one (k15) formation containing its in the K area of the triangle. The SEM photograph of **Figure 9** shows some part of the K area containing k1, k13, k15, k16 and k17. Other neighbouring particles in that area are : k3-5, a Coccolith ; k6, a PVC plastic ; k7, a lapis lazuli; k8, a calcium carbonate ; k10, a Tintinoid ; k12, a Dinophycae ; k14, an organic fragment.

Figure 9. The k1 and k13 particles. *Above*. SEM photograph (2 000x), in BSE, of the upper part of the K area showing particles k1 and k13 (and formation k15 and particles k16 and k17). *Below*. The k1 (Lower spectrum) and the k13 (upper spectrum) spectras (insert above the k13 spectrum indicates the Cu/Zn proportions).

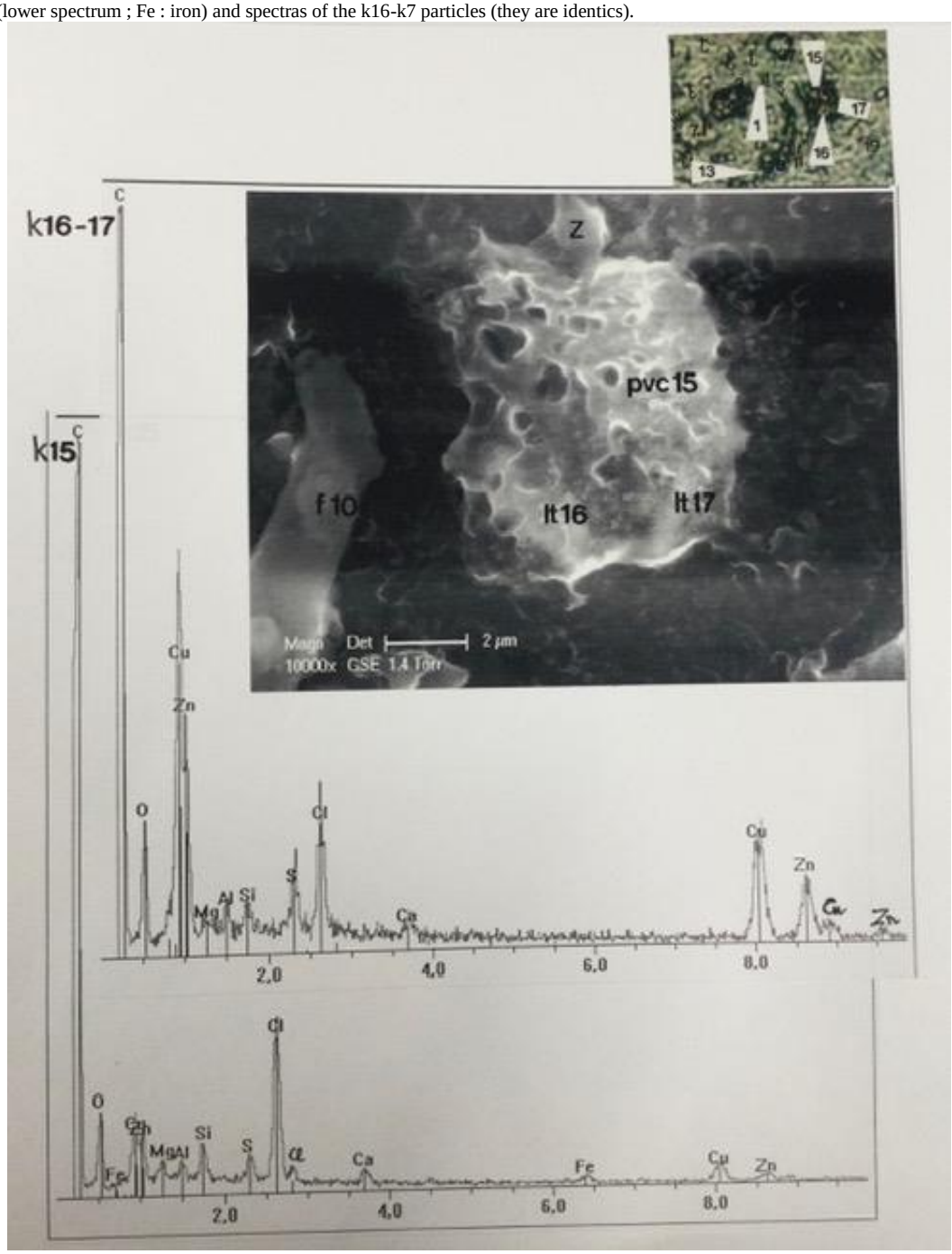


The k1 particle is a micro-sheet, of about 1.7 µm of length. In its spectrum, the Cu proportion is lightly superior to that of the Zn (the Cu>Zn type). Its colour is black in optic microscopy.

The k13 particle is a micro-sheet, of about 1.5 µm of length. In its spectrum the Cu proportion is also lightly superior to that of the Zn (semi-quantitative measures showing the proportions of 54.6 % of Cu and of 45.6% of Zn), so it is of the Cu>Zn type. Its colour is clear-yellow in optic microscopy.

The SEM photograph of **Figure 10** shows the k15 formation. It is a squared sheet, multi-perforated, of about 9.5 µm of side. The k16 and k17 particles are located on its inferior side and a small particle (the z particle, of about 2µm of size, and that is a zinc oxyde) at its superior border. In the k15 spectrum, rich in chlorine (Cl), the Cu and Zn proportions are equal (of the Cu=Zn type). The k15 colour is yellow in optic microscopy.

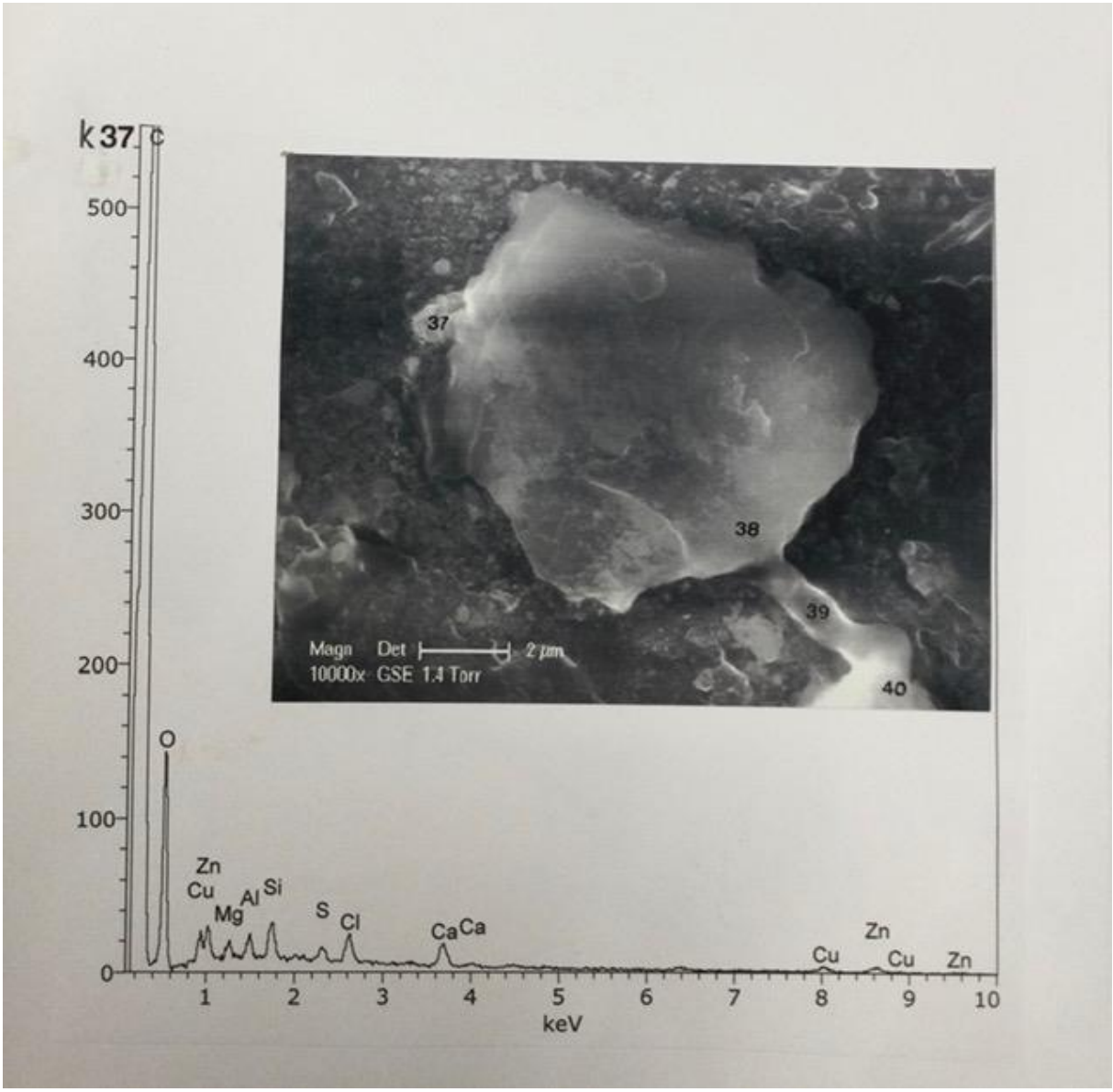
Figure 10. The k15 formation and the k16 and k17 particles. *Lower photograph*. SEM photograph (10 000x), in GSE, showing the k15 (pvc 15) formation and the k16 and 17 (lt16 and lt17) particles (Z is a zinc oxyde ; f10 is the vertical extremity of the fourth linen fiber). *Upper photograph.* Photograph in optic microscopy (1 200x) of the K area part showing k1, k13, k15, k16, and k17. *Below*. Spectrum of the k15 formation (lower spectrum ; Fe : iron) and spectras of the k16-k7 particles (they are identics).



The k17 particle is a micro-sheet, of about 2.2 µm of length. In its spectrum the brass is also of the Cu : 2/Zn : 1 type. Its colour is also yellow-red in optic microscopy.

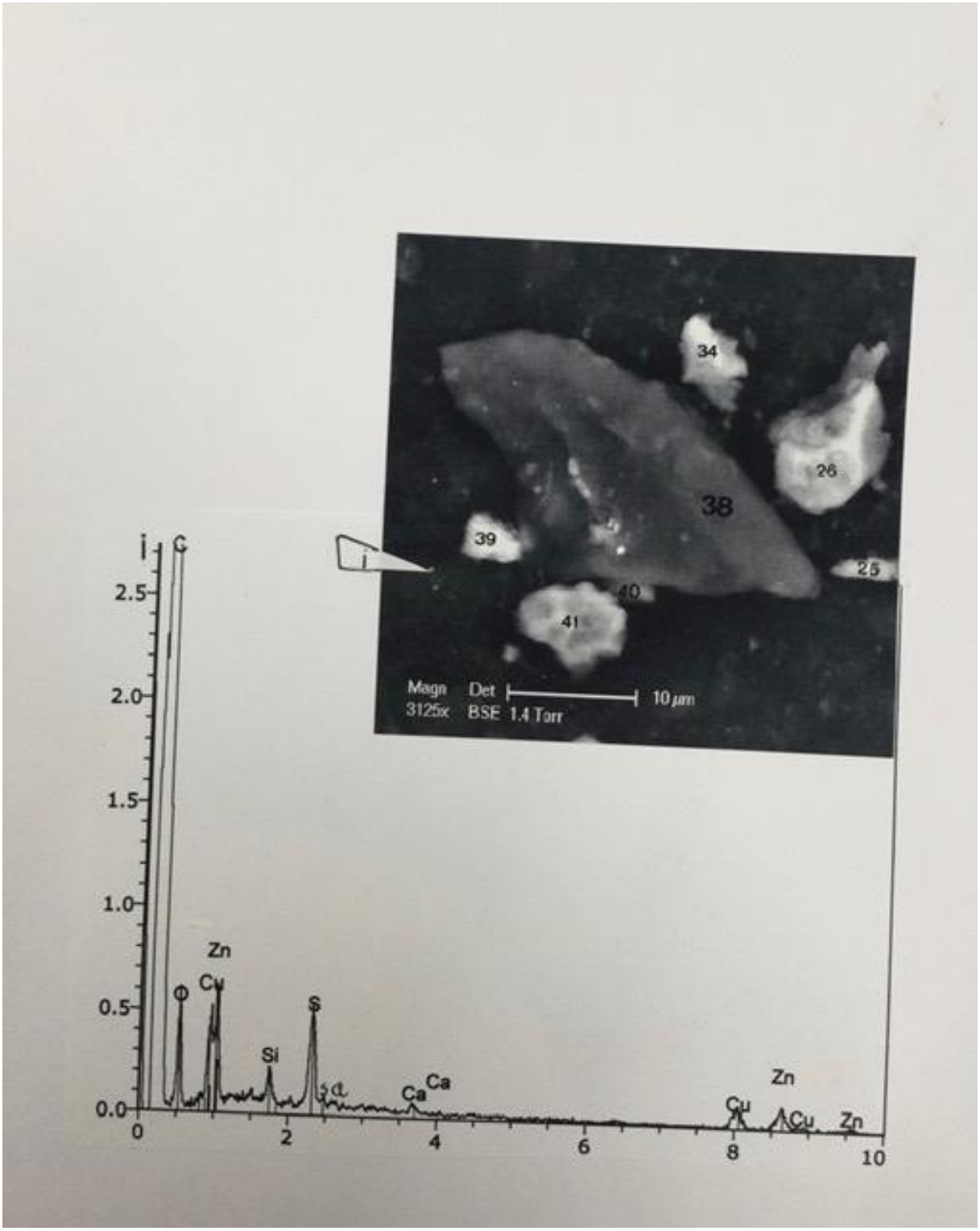
The SEM photograph of **Figure 11** shows the k37 particle ; it is located near the left side of k38 (a stained-glass window fragment) ; other neighbouring particles are : k39, an aragonite and k40, a calcium carbonate. The k37 particle is a micro-ball, with a diameter of about 0.9 µm. In its spectrum the Cu proportion is approximatively the same than that of Zn (the Cu=Zn type). The k37 colour is yellow-red in optic microscopy.

Figure 11. The k37 particle. *Above*. SEM photograph (10 000x), in GSE of some lower part of the K area showing k37. *Below*. The k37 HR spectrum.



The SEM photograph of **Figure 12** shows some part of the L area containing the i particle (located on the left side of l39). Other neigbouring particles in that area are : l25, a talcum ; l26, an alumina-silicate iron-rich ; l34, a calcium phosphate ; l38, a wax ; l39, an hematite [**8**] ; l40, a calcium carbonate ; l41, a quartz.

Figure 12. The i particle. *Above*. SEM photograph (3 125x), in BSE, of the central part of the L area showing the i particle. *Below*. The i HR spectrum.



The i particle is a micro-ball, of about 0.5 µm of diameter. In its spectrum, this brass is of the Cu=Zn type. Its colour is yellow-red in optic microscopy.

There are three brass particles, closely located, on the M area : m62, m63 and m64. The SEM photographs of **Figure 13** show some part of the M area, with m62-64 locations. The three are co-located on a triangular formation of about 5 µm of size, of organic matter, rich in chlorine and of green colour in optic microscopy. **Figure 14** shows m62 and m63 spectras.

Figure 13. The m62, m63 and m64 particles. The upper photograph is a SEM view (16 000x), in GSE, of these three particles and of the organic triangle on which they are located. The lower photograph is another SEM view (24 000x), in BSE, of the particles.

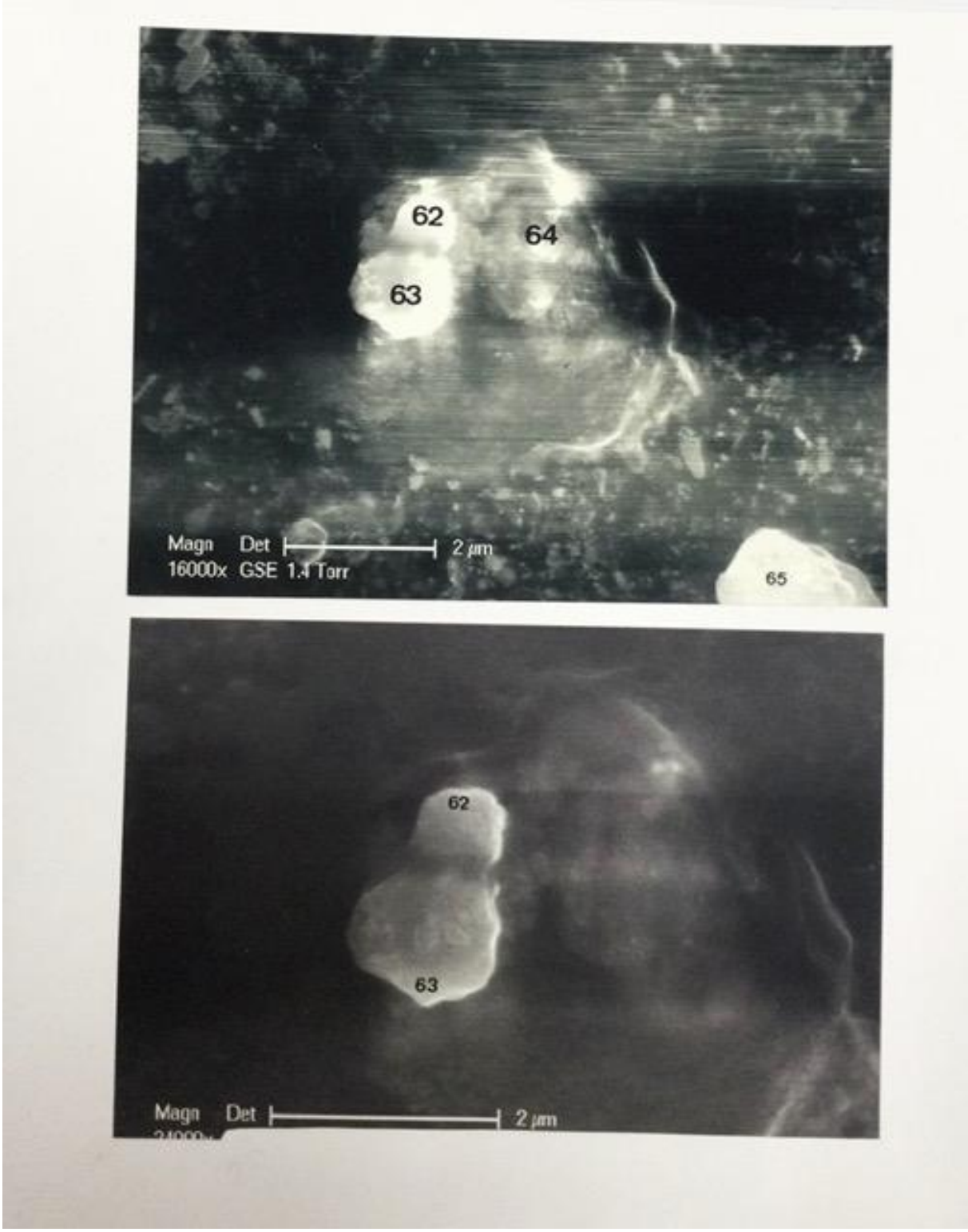
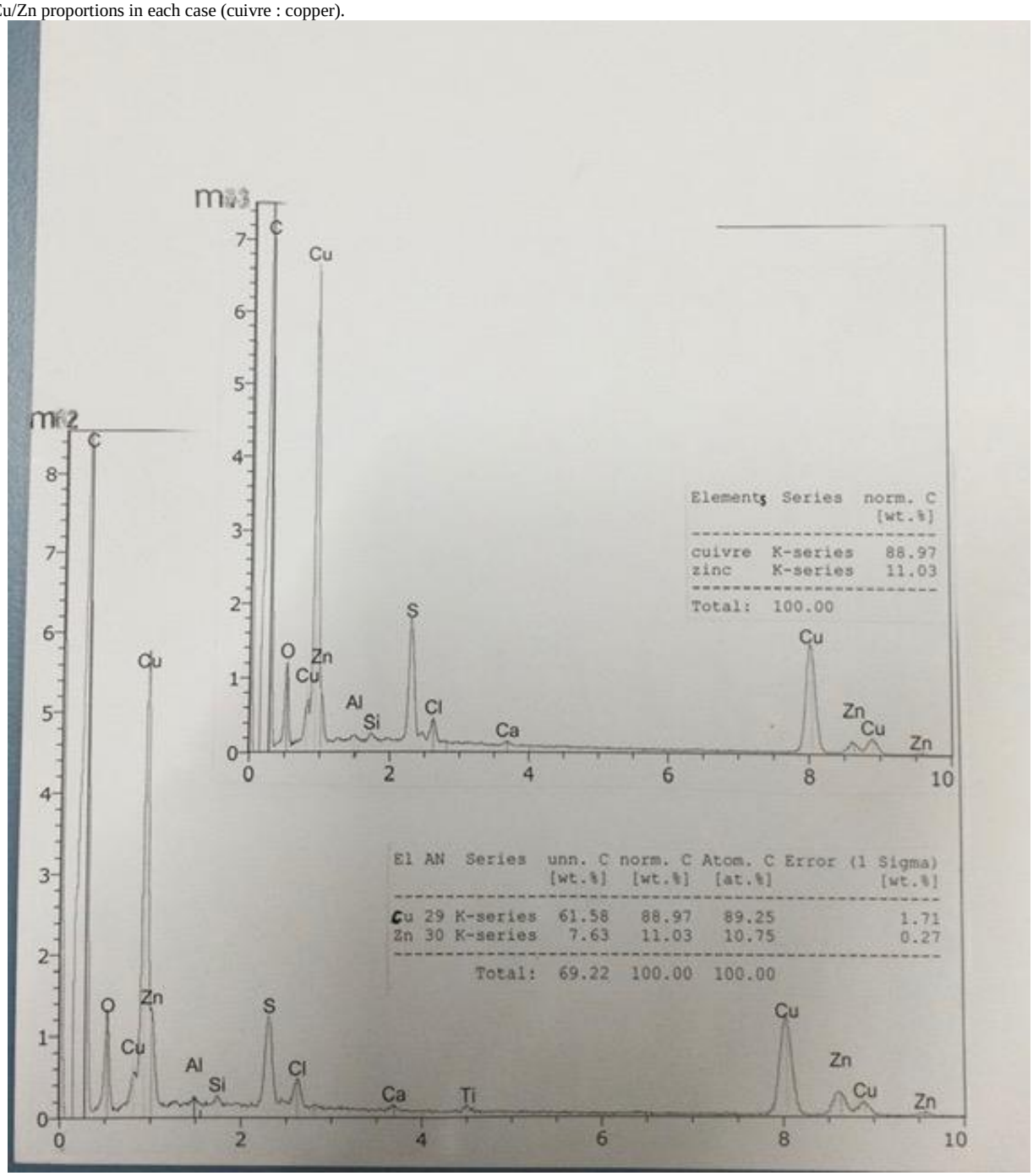


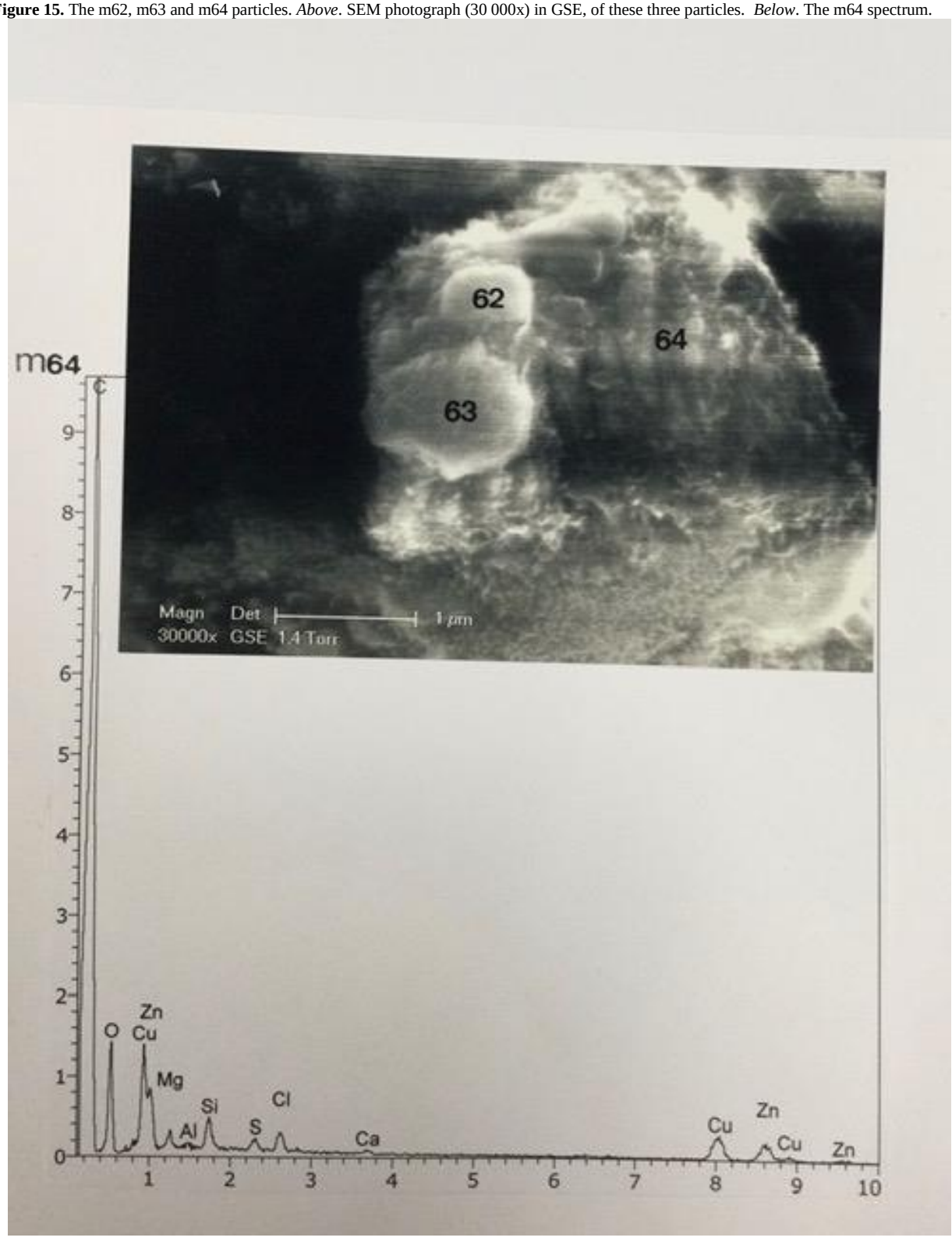
Figure 14. Spectras of the m62 and m63 particles. *Lower spectrum*. That of m62 (Ti : titanium). *Upper spectrum*. That of m63. Inserts indicates Cu/Zn proportions in each case (cuivre : copper).



The m62 particle is a micro-ball, of an approximate diameter of 0.9 µm. its spectrum shows proportions of Cu of 88.9% and of Zn of 11.1% ; it is of the Cu>>>Zn type. Its colour is green in optic microscopy. The m63 particle is a micro-ball, of an approximate diameter of 1.5 µm. Its spectrum shows the same proportions of Cu and Zn (it is also of the Cu>>>Zn type). Its colour is also green in optic microscopy.

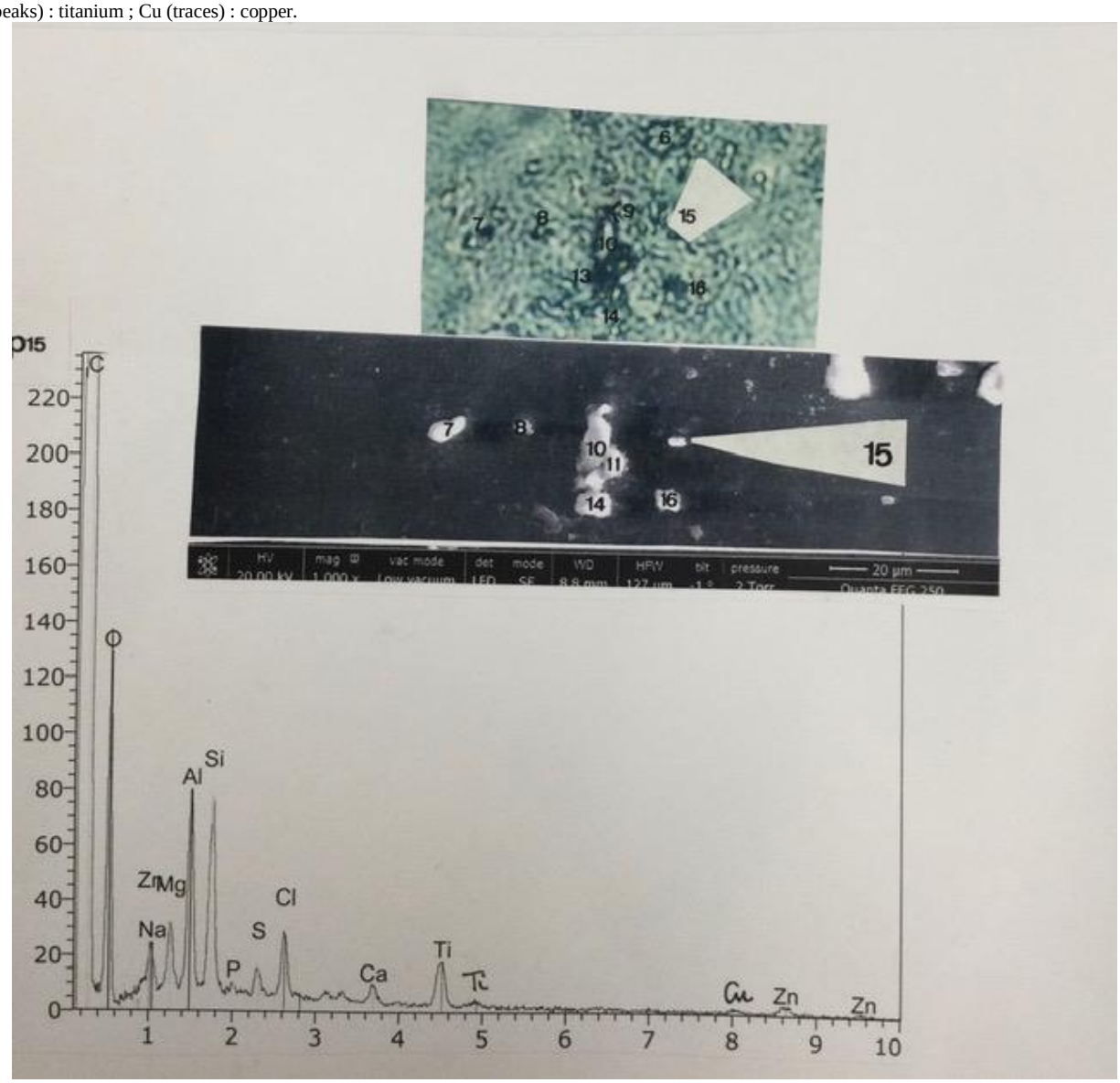
The SEM photograph of **Figure 15** shows another GSE view of the little organic triangle where m62 and m63 particles are located ; the m64 particle, hidden at the interior of the triangle, disappears. The m64 particle is a micro-sheet, of about 2.5µm of length. In its spectrum the Cu proportion is approximatively double than that of Zn (so, of the Cu : 2/Zn : 1 type). Its colour is black in optic microscopy.

Figure 15. The m62, m63 and m64 particles. *Above*. SEM photograph (30 000x) in GSE, of these three particles. *Below*. The m64 spectrum.



There is not brass particles in areas N and O. The SEM2 photograph of **Figure 16** shows some part of the area P with the p15 formation containing brass. Other neighbouring particles in that area are : p7, a marble fragment ; p8, a Cyanophycae ; p10, an alumina-silicate of potassium with iron ; p11, a lapis lazuli ; p14, a calcium phosphate ; p16, a silicate.

Figure 16. The p15 formation. *Lower photograph*. SEM2 photograph (1 000x), in LFD, of the central part of the P area showing p15. *Upper photograph*. Photograph in optic microscopy (1 200x) of this area part. *Below*. The p15 spectrum. C : carbon ; O : oxygen ; Na : sodium ; Zn (three peaks) : zinc ; Mg : magnesium ; Al : aluminium ; Si : silicium ; P : phosphorous (traces) ; S : sulphur ; Cl : chlorine ; Ca : calcium ; Ti (two peaks) : titanium ; Cu (traces) : copper.



The p15 formation is an alumina-silicate, titanium (Ti)-rich. It is a micro-sheet of about 3.5 µm of length. In its spectrum, the Zn proportion is very more elevated than that of Cu, which is present in the form of traces (of the type Zn>>Cu). The p15 colour is clear-yellow in optic microscopy.

There is not brass particle in area Q. The area R contains two brass particles : r15 and r22. The SEM photograph of Figure 17 shows some part of the R area containing r15. Other neighbouring particles in that area are : r11, a PVC plastic ; r16, that is a part of this PVC plastic ; r24, a gypsum.

The r15 particle is a micro-ball, of about 2.5 µm of diameter. In its spectrum the Cu/Zn proportions are of the Cu : 2/Zn : 1 type. Its colour is orange in optic microscopy.

The SEM photograph of **Figure 18** shows, in a lower portion of the R area, the r22 brass particle. It is a micro-sheet, of about 3.2µm of length. In its spectrum the Cu proportion is of 38.7 % and the Zn proportion of 61.3% ; it is of the Zn>Cu type. The r22 colour is clear-yellow in optic microscopy.

Figure 18. The r15 and r22 particles. *Above*. SEM photograph (600x), in BSE, of the R area showing the full length of r11 ; locations of the r15 and r22 particles are indicated (**β** is the right border of the triangle). *Below*. The r22 HD spectrum (insert indicates the Cu/Zn proportions).

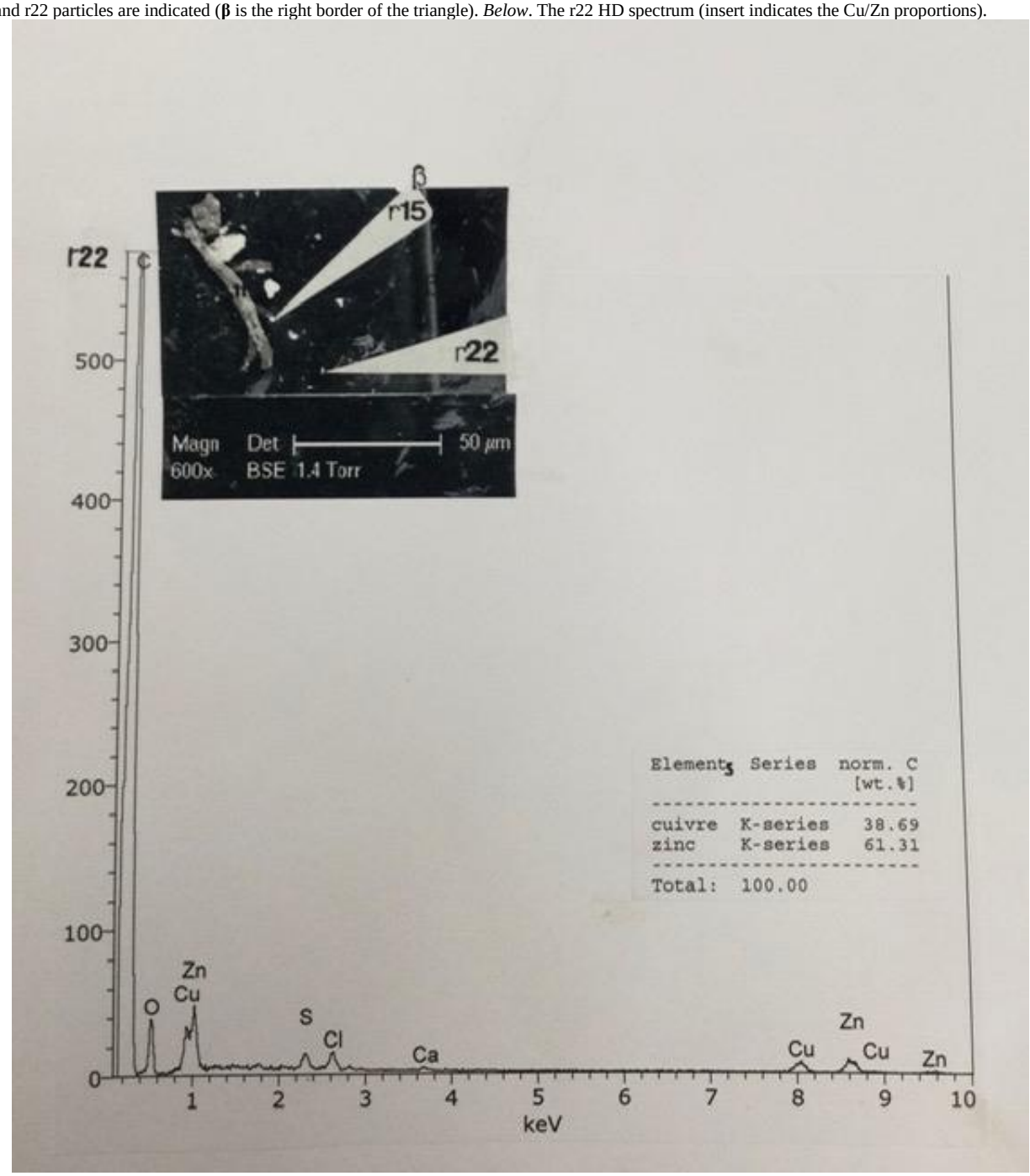
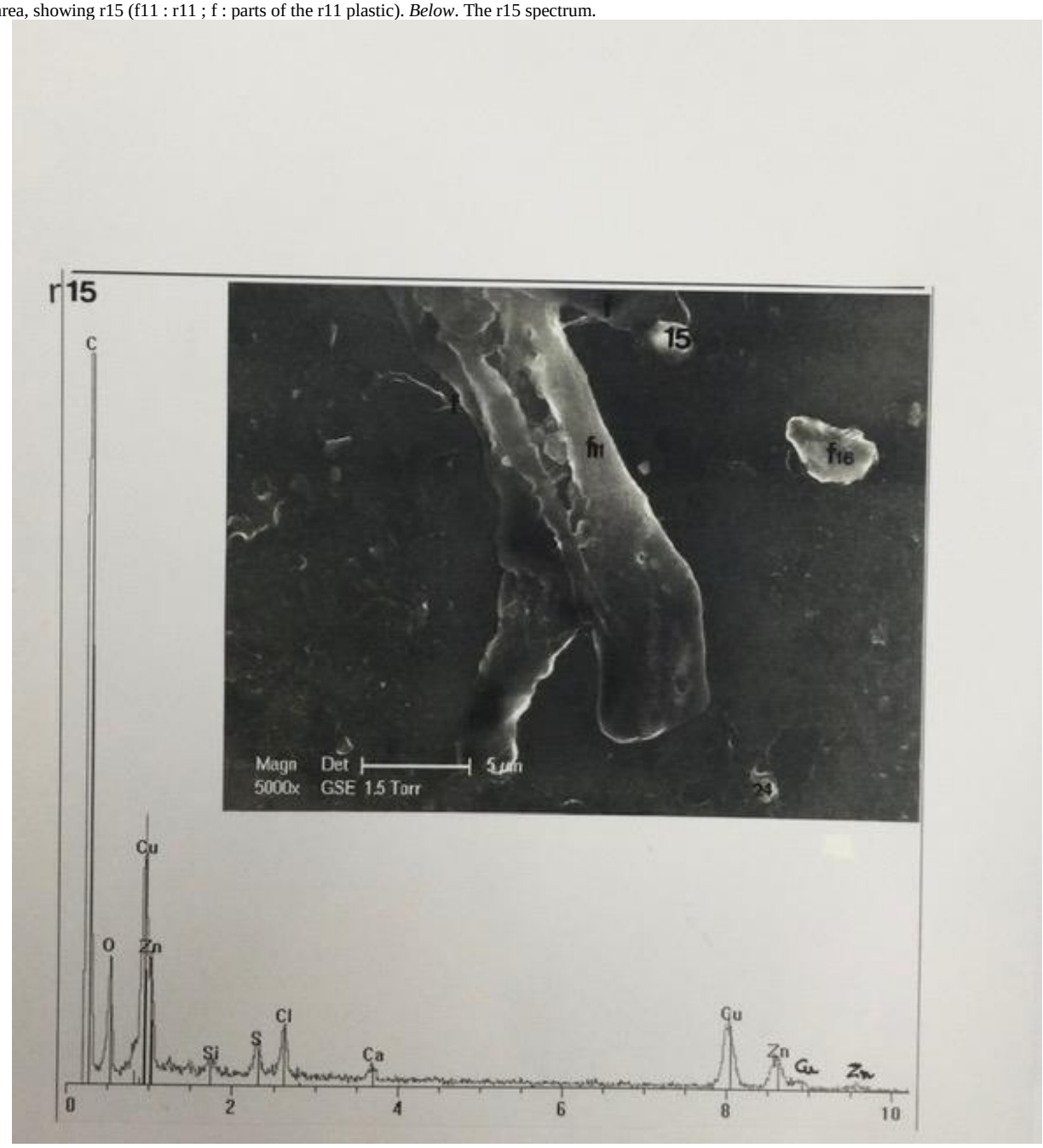
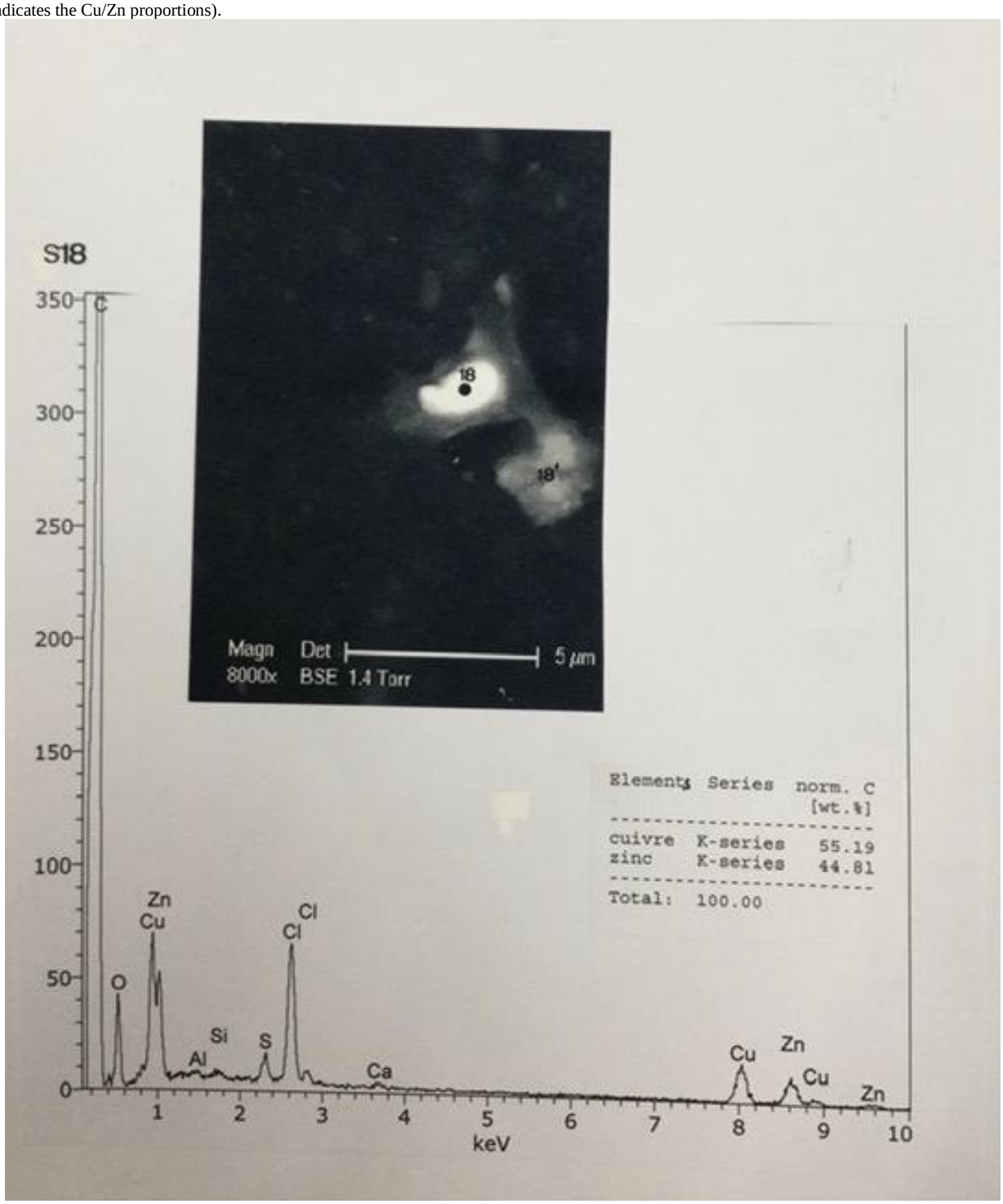


Figure 17. The r15 particle. *Above*. SEM photograph (5 000x), in GSE, of the lower part of the r11 PVC plastic located in the low part of the R area, showing r15 (f11 : r11 ; f : parts of the r11 plastic). *Below*. The r15 spectrum.



The SEM photograph of **Figure 19** shows, in some part of the S area, the s18 brass particle. Both s18 and s18' (a gypsum) are included in an organic matter Cl-rich. The s18 particle is a micro-sheet, of about 1.9 µm of length. In its spectrum the Cu proportion is of 55.2% and the Zn proportion of 44.8%, so it is of the Cu>Zn type. The s18 colour is yellow in optic microscopy.

Figure 19. The s18 particle. *Above*. SEM photograph (8 000x), in BSE, showing s18 (and s18') in the S area. *Below*. The s18 HR spectrum (insert indicates the Cu/Zn proportions).



4. Discussion

Table 1 lists and characterizes the twenty-two (one in the B area **:** b53 ; three in the C area : c1, c7 and c33 ; three in the E area : e43, l and e130 ; one in the F area : f9 ; six in the K area : k1, k13, k15, k16, k17 and k37 ; one in the L area : i ; three in the M area : m62, m63 and m64 ; one in the P area ; p15 ; two in the R area : r15 and r22 ; one in the S area : s18) brass particles and formations containing its detected on the various areas of the triangle. It appears that there is a local concentration (27.3%) of brasses in the K area.

Table 1 : Nomenclature and characterization of the twenty-two brass particles found on the triangle.

Nomenclature	Areas	Particles	Forms	Typical spectras	Cu/Zn compositions	Peculiarities
1	B	b53	ovale sheet	+	Cu=Zn	with aluminium
2	C	c1	micro-sheet	+	Cu:2/Zn:1	
3	C	c7	micro-sheet	+	Cu:2/Zn:1	
4	C	C33	micro-ball	+	Cu=Zn	
5	E.c	e43	micro-sheet	+	Cu>Zn	
6	E.g	l	micro-sheet	+	Cu>>Zn	on e57
7	E.g	e130	micro-ball	+	Cu>>Zn Cu (70%) ; Zn (30%)	very deteriorated
8	F	f9	micro-ball	+	Cu:2/Zn:1	
9	K	k1	micro-sheet	+	Cu>Zn	
10	K	k13	micro-sheet	+	Cu>Zn ; Cu (54.6%) ; Zn (45.4%)	
11	K	k15	squared sheet	+	Cu=Zn	
12	K	k16	micro-sheet	+	Cu:2/Zn:1	on k15
13	K	k17	micro-sheet	+	Cu:2/Zn:1	on k15
14	K	k37	micro-ball	+	Cu=Zn	
15	L	i	micro-ball	+	Cu=Zn	
16	M	m62	micro-ball	+	Cu>>>Zn ; Cu (88.9% ; Zn (11.1%)	on a triangular sheet
17	M	m63	micro-ball	+	Cu>>>Zn ; Cu (88.9% ; Zn (11.1%)	on the triangular sheet
18	M	m64	micro-sheet	+	Cu:2/Zn:1	on the triangular sheet
19	P	p 15	micro-sheet	a silicate	Zn>>Cu (traces)	with titanium excess
20	R	r15	micro-ball	+	Cu:2/Zn:1	
21	R	r22	micro-sheet	+	Zn>Cu ; Cu (38.7%); Zn (61.3%)	
22	**S**	s18	micro-sheet	+	Cu>Zn ; Cu (55.2%); Zn (44.8%)	coated with s18', in a sheet Cl-rich

The three formations (b53, k15 and p15) containing brass are distinguished from the brass particles, that are micro-sheets (c1, c7, e43, l, k1, k13, k16, k17, m64, r22, s18) or micro-balls (c33, e130, f9, k37, I, m62, m63, r15).

The distribution of sizes among the brass particles varies between 0.5 µm (particle 15) and 7.2 µm (particle 2), with a model class of 1.7 to 2.5 µm (particles 4, 5, 7, 8, 9, 13, 18, 20, 22).

The b53 spectrum contains a very elevated proportion of the aluminium element ; this formation corresponds probably to a modern recycled aluminium fragment with brass.

We have not found any brass particles with lead, tin, nickel, chromium or arsenic.

The Cu/Zn proportions vary greatly among the different brasses detected.

For simplification, they are lumped in four main categories : the first category is that where these proportions correspond to Cu : 2/Zn : 1 ; the second includes all other proportions where Cu is superior to ZN (Cu>Zn ; Cu>>Zn ; Cu>>>Zn) ; the third is that where Cu=Zn, and the fourth includes proportions where Zn>Cu. **Table 2** recapitulates brass numbers belonging to each category.

Table 2 : The four categories of brasses found.

Categories	**Cu/Zn nomenclatures**	**Brass numbers**
1	Cu:2/Zn:1	2, 3, 8, 12, 13, 18, 20, 22
2	Cu superior to Zn	5, 6, 7, 9, 10, 16, 17
3	Cu=Zn	1, 4, 11, 14, 15
4	Zn>Cu	19, 21

There are three sorts of brasses regarding to colour : “red brasses” with Cu>Zn values, “white brasses” with Zn>Cu values and “yellow brasses” for intermediate values.

Table 3 relates colours of the twenty-two brasses according to Cu/Zn categories. To explain some colours are taken in consideration : brass sizes, S proportions (measured as peak height of sulphur compared to that of the Cu Kα1 ray) to estimate the black corrodation, and Cl proportions (measured as peak height of chlorine compared to that of the Cu Lα1 ray) to estimate the contamination by the green organic material.

Table 3 : Colours of the brasses observed in optical microscopy.

Brasses numbers	**Sizes (in µm)**	**Cu/Zn categories**	**Cl proportions**	**S proportions**	**Colours**
1	13.5	3	Cl>>Cu	S>Cu	yellow
2	7.2	1	Cl<Cu	S<Cu	yellow-red
3	3.5	1	Cl<Cu	S>Cu	black
4	2.2	3	Cl<Cu	S=Cu	yellow
5	2.1	2	Cl<Cu	S>Cu	yellow
6	0.9	2	Cl<Cu	S>Cu	black
7	2.3	2	Cl<Cu	S=Cu	yellow
8	1.9	1	Cl<Cu	S=Cu	yellow
9	1.7	2	Cl<Cu	S>Cu	black
10	1.5	2	Cl<Cu	S=Cu	clear-yellow
11	9.5	3	Cl>Cu	S>Cu	yellow
12	3.2	1	Cl<Cu	S<Cu	yellow-red
13	2.2	1	Cl<Cu	S<Cu	yellow-red
14	0.9	3	Cl=Cu	S>Cu	yellow-red
15	0.5	3	Cl (traces)	S>Cu	yellow-red
16	0.9	2	Cl<Cu	S=Cu	green
17	1.5	2	Cl<Cu	S=Cu	green
18	2.5	1	Cl<Cu	S<Cu	black
19	3.5	4	Cl>>Cu	S>Cu (traces)	clear-yellow
20	2.5	1	Cl<Cu	S<Cu	orange
21	3.2	4	Cl<Cu	S=Cu	clear-yellow
22	1.9	1	Cl=Cu	S<Cu	yellow

Colours of the brass particles are generally yellow : yellow-red in brassed numbers 2, 12 and 13, and orange in brass number 20 (all of the first category), clear-yellow in brasses numbers 19 and 21 (of the fourth category). But brasses numbers 8 and 12 (of the first category) are yellow. Brasses numbers 5 and 7 (of the second category) are also yellow, as brasses numbers 1, 4 and 11 (of the third category) ; but brasses numbers 14 and 15 (also of the third category) are yellow-red, and brass number 10 (of the second category) is clear-yellow.

Brasses numbers 3 and 9, of black colour, are corroded (S>Cu Kα1) ; corroded also is the brass

number 6, of black colour, which is of little size (< 1µm).

Brasses m62 (number 16), m63 (number 17) and m64 (number 18) depict a special feature : brasses numbers 16 and 17 are green, as the colour of the little organic Cl-rich triangular matter where they are located ; brass number 18, located inside of this triangular matter, is black. The whole constitutes probably a triangular painting fragment with brass.

5. Conclusions.

In the present study, a total number of twenty-two brasses were detected on the triangle surface. The form of the nineteen brass particles detected are elongated (the micro-sheets) or rounded (the micro-balls), and their sizes vary between 0.5 and 7.2 µm (with a modal class of 1.7-2.5 µm).

Copper/zinc proportions varies greatly among the different brass particles detected. Four categories of Cu/Zn composition are considered here : the first corresponds to Cu : 2/Zn : 1 ; the second includes all other proportions where Cu is superior to Zn ; the third is that where Cu=Zn and the fourth is that where Zn>Cu. The percentages of copper in the alloys (compared to those of zinc) are of 88.9 % in particles 16-17, of 70% in particle 7, of about 66% in particles 2, 3, 8, 12, 13, 18 and 20, of about 55% in particles 10 and 22, of about 50% in particles 4, 14 and 15, and of 38.7 % in particle 21. Such a diversity in the Cu/Zn compositions suggest that the Turin Shroud was sprinkled with different brass powders during history.

Colours of the brass particles detected are generally yellow. Typically yellow-red or yellow in brass particles of the first and second category, and clear yellow in brass particles of the fourth category (black particles are those where the Cu of the alloy is corroded by sulphur). So these particles, concerning the yellow colour, imitate gold. Among them the brass particle number 21 (which is clear-yellow) corresponds to that sort of white brass alloy that was reserved for prestigious objects, as those of aristocratic and lithurgic cockeries.

Some forms of brass have been in use since prehistory. The earliest brasses may have been natural alloys made by melting zinc-rich copper ores. By the Roman period, brass was produced from metallic copper and zinc minerals (such as the calamines), using the cementation process, and variations of this method continued until mid-19th century. At the end of the medieval period brass were very successful, because its yellow and sparkling colour that remains gold. Cementation was replaced by speltering (the direct allowing of copper and zinc metal), which was introduced to Europe in the 16th century.

Formation p15 is representative of the cementation process of brass production. It is a micro-sheet of 3.5 µm of length, consisting of a calamine (a zinc ore), which is a peculiar alumina-silicate (titanium-rich) with zinc metal. It contains traces of (exogenous) copper.

The formation k15 is representative of the direct process of brass production. It is a squared sheet of 9.5 µm of side, consisting of a brass alloy of Cu=Zn composition. Its porous aspect reveals the elevated temperature of the process. In its superior side is the z particle, that is a zinc oxide, and two brass particles (k16 and k17, of identic compositions) are loaded on its inferior face.

Some fine grains of a gold powder were deposited on the surface of the Turin Shroud [**9**]. We propose that the brass particles reported here were loaded on the T.S. surface , at different times, to imitate gold. A fine brass powder is again proposed to-day-for example CM4357 Spherical Brass Alloy (Cu Zn 4) Powder-for decorative applications.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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