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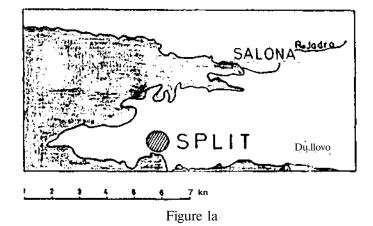
# CERAMICS FROM THE ROMAN CITY OF SALONA NEAR SPLIT

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Ceramics from the Roman city of Salona were examined by optical microscopy, X-ray diffraction, and by micropaleontological and chemical methods. It was concluded that a portion of the ceramics voas made of the raw material from the sediments of Eocene flysch in the immediate surroundings of Salona, and that the firing temperatures ivere betvueen 800" and 850"C.

#### Introduction

The Roman city of *Salona* near Split (Fig. 1) had the status of a Roman colony and was especially prosperous in the time of the emperors Trajan, Hadrian, Antoninus Pius and Diocletian. The urban nucleus of *Salona* with ports, walls, forum, theatres, temples, baths and private villas was formed in the 2nd century A. D. The large amphitheatre from the 2nd century was incoporated into the town fortification system.



Archaeological studies in the area of *Salona*, along the newly built Splitbypass, were carried out in 1986 and 1987. On that occasion Roman architecture was discovered at the locations of the *»Ancient street«* and the *Hortus Metrodori* necropolis, Many of movable archaeological objects were also found. Teams from the Croatian Restoration Institute from Zagreb and the Archaeological Museum from Split collected samples of

stones, mortar and ceramics, before the site was covered again to allow the construction of the bypass. Part of the samples collected was analyzed by optical microscopy, paleon-tological, X-ray diffraction and chemical methods.

The present paper describes preliminary results of the examination of ceramic samples. Special attention was paid to the origin of the raw material used in ceramics preparation and the firing temperature of the ceramics.

The samples analyzed were marked RZH-2146 and RZH-2147. They were taken at the site of the *»Ancient street«*. The sample RZH-2146 was taken from a dike on the northern side of the street, while the sample RZH-2147 was found in the southern fork of the street under a well preserved antique mosaic (from Malinar and Smailagić, 1987).

### Methods of analysis

The ceramic samples were examined by optical microscopy, X-ray diffraction and by chemical methods.

A polarizing microscope was used to determine the composition of the ceramics and the microorganisms contained in the samples.

X-ray powder diffraction was used in order to determine the mineral composition of the ceramics, and the composition of the supposed raw material from the immediate surrounding of Salona, clay sediment of the Eocene flysch from the locality of Duilovo (Fig. 1). The flysch from Duilovo was used to prepare a composite material in a simulation procedure of the ceramics firing. The composite material and the antique ceramics RZH-2146 were fired at a series of temperatures. The phase composition of the fired samples was found for each temperature, and on that basis the firing temperature of the Salona ceramics was determined. X-ray powder diffraction patterns were taken by means of the counter diffractometer, with CuK *a* radiation. The minerals were denoted in diffraction patterns as follows (Figs. 4-6):

- I illite
- Q quartz
- P plagioclase
- K potassium feldspar
- Ca calcite
- C calcium oxide
- H hematite
- Ge gehlenite
- D diopside

The chemical composition of one ceramics and of the insoluble residue of the flysch were determined by the classical quantitative chemical silicate analysis.

### **Results of examinations**

Two ceramic samples were examined.

The first sample from ceramics RZH-2146 was 6 to 7 mm in thickness and was half red and half yellow in colour, with smooth surfaces on both sides.

The second sample from ceramics RZH-2147 was 5 to 7 mm in thickness, and was red throughout, with a furrowed convex part so that it resembled a furrowed shell.

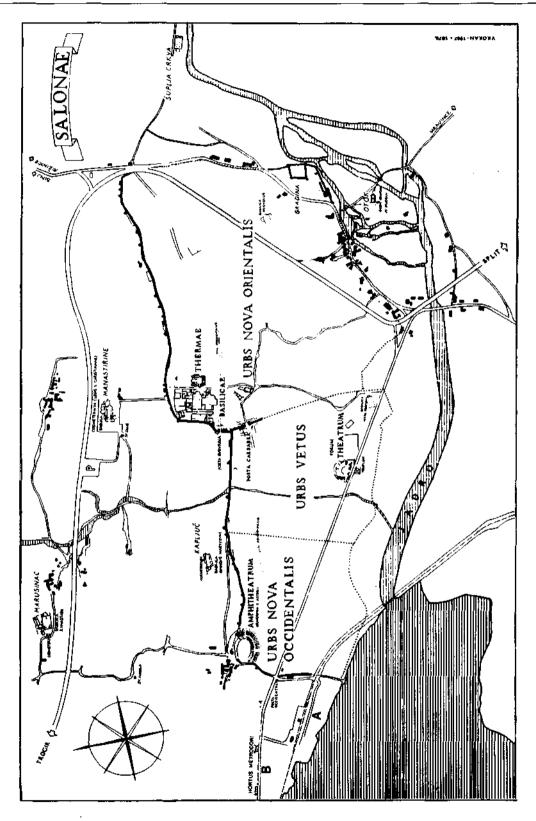


Figure 1b

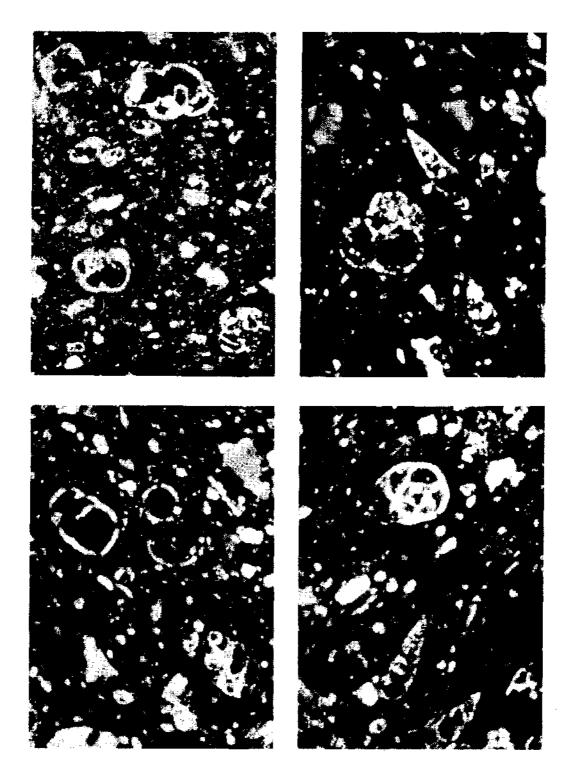


Figure 2

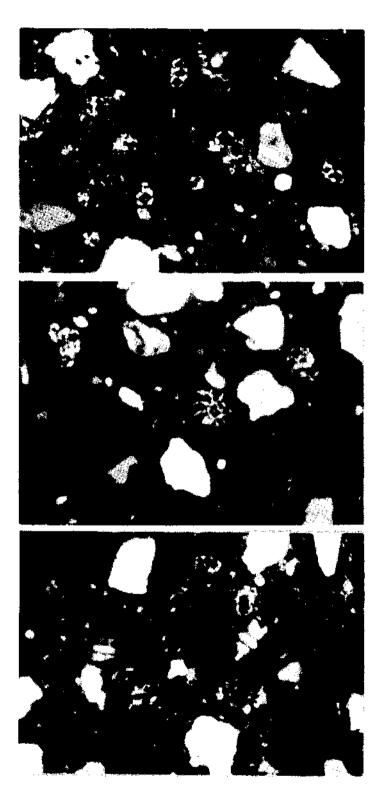


Figure 3

#### Microscopic analyses

Thin sections of the ceramic sample RZH-2146 from two differently coloured parts, did not show a difference in the mineral composition, but only in the appearance of the microscopically indeterminable matrix. Anisotropic effects were slightly visible in the matrix of the red part, whereas the matrix of the yellow part was vague and partially non-transparent.

The ceramics contained the following microscopically determined components: an abundance of well preserved calcite skeletons of foraminiferes (Fig. 2), particles of quartz and an amount of quartzite, chalcedonic chert, feldspars, microscopically minute calcite irregular aggregates, muscovite-sericite, epidote, an opâque mineral and tourmaline.

The dimensions of the components were as follows: for a minifers having the diameter up to 0.25 mm, quartz particles of the diameter up to 0.22 mm, grains having in sections up to 0.24 x 0.32 mm<sup>2</sup>, quartzite particles with the diameter up to 0.28 mm, muscovite lamina uptoO.02xO.15 mm<sup>2</sup>, and the aggregates of fine-grained calcite up to 0.25 x 0.30 mm<sup>2</sup>.

The skeletons of the foraminifers, made of calcite, were thin with central parts porous and empty. Quartz grains were angular in shape, or they were cracked. Quartzite had a granoblastic texture, while tourmaline was clearly pleochroitic.

A thin section of ceramics RZH-2147 contained the same components as the sample RZH-2146 described above (Fig. 3). The skeletons of foraminifers in this sample were not well preserved and were partly distorted. Quartz grains of diameters from 0.15 to 0.38 mm, were angular in shape, or partly cracked. The ceramics also contained small fractions of feldspars (microcline), quartzite, chalcedonic chert and tourmaline, as well as aggregates of microcrystalline calcite.

The calcite skeletons in the ceramics RZH-2146 were represented by the following foraminifers:

Globigerina yeguensis (WEINZIERL & APPLIN) Globigerina sp. div.

Globorotalia sp.

Globigerinatheca ex gr. mexicana (CUSHMAN)

G. mexicana kugleri (BOLLI, LOEBLICH & TAPPAN)

Turborotalia cerroazulensis frontosa (SUBBOTINA)

T. cerroazulensis premoli (TOUMARKINE & BOLLI)

? Bulimina sp.

Nodosaridae

The stratigraphic affiliation of the foraminifers can be stated as Middle Eocene (Lutetium).

The calcite skeletons in the ceramics RZH-2147 were represented by the following foraminifer genuses:

Globorotalia Globigerina Pseudohastigerina ? Globigerinatheca

### X-ray diffraction and chemical analysis

The ceramics RZH-2146 was analysed chemically (Table 1) and by X-ray powder diffraction. The diffraction patterns were taken of the as-found (original) sample, and the ceramic sample treated by 1.5% acetic acid for 10 minutes (Fig. 4). It was found that the ceramics contained calcite (22%), quartz (15%), plagioclase (5%), and small amounts of illite (or muscovite), hematite, potassium feldspar, gehlenite, diopside and an amorphous fraction. The ceramics probably contained also a fraction of structurally unidentified calcium-aluminium-silicate hydrates; this conclusion was based on the weight loss (of 6.17%) during heating up to 600°C (Table 1). Bulk pieces of the ceramics (not the powder) were baked at 750°C, 800°C and 900°C for 3 hours. According to the powder diffraction patterns of these samples (Fig. 5), diffraction line intensities of gehlenite and diopside (characteristic silicate minerals in ceramics) increased with the increase of temperature. Diffraction patterns of the fired samples did not contain diffraction lines of calcite. Calcite was decomposed at a temperature below 750°C; the calcite of foraminifers transformed mainly into calcium oxide, while the dispersed calcite, with fine grains, reacted with silicates forming calcium silicates.

A sample of the Eocene flysch from the locality of Duilovo was selected, containing  $\sim 45\%$  of calcite. The calcite in this sample was solved in 1.5% acetic acid, and the insoluble residue was analysed chemically (Table 1) and by X-ray powder diffraction. The approximate mineral composition of the insoluble residue was as follows (weight percent): 25% of illite, 23% of quartz, much of irregularly interstratified phyllosilicates and an amorphous fraction, 20% of smectite and vermiculite, 6% of plagioclase, and small amount of chlorite and potassium feldspar. One can see from Table 2 that the ratios of chemical components (oxides) in the ceramics and flysch were rather similar. Therefore, the insoluble residue of flysch could be used to obtain the composite material in a simulation procedure of the ceramics preparation. The amount of calcite in the raw material used in preparation of the antique ceramics, according to the data for calcium in Table 1, was  $\sim 30\%$ . Therefore, the composite material was prepared to have the following composition (weight percent): 70% of the insoluble residue, 20% of powdered calcite and 10% of the calcitic Eocene foraminifers (grains having 0.3 to 0.2 mm in diameter). The composite material was fired for 3 hours at 750, 800, 850 and 900°C and X-ray powder diffraction patterns of these samples were taken. The sample fired at 750°C did not any more contain calcite. X-ray diffraction pattern of the sample fired at 800°C contained weak diffraction lines of gehlenite and diopside. Diffraction lines of gehlenite and diopside became stronger and well-defined as the firing temperature incrased. Diffraction pattern of the sample fired at 850°C still contained diffraction line of illite at  $0 \sim 10^{\circ}$ , but the structure of illite was destroyed after firing at 900°C.

Diffraction patterns of the composite material and of the ceramics RZH-2146 fired at the same temperatures were almost identical. These two samples possessed a very similar mineral composition after the same thermal treatment. For instance, both samples when fired at 900°C (Fig. 6) had the following mineral composition: quartz, gehlenite, diopside, calcium oxide, plagioclase, hematite, potassium feldspar and an amorphous fraction.

X-ray powder diffraction was also used to analyse the ceramics RZH-2147. The

mineral composition of this ceramics was different from that of the ceramics RZH-2146. The following minerals were identified in RZH-2147: 36% of quartz, 20% of calcite, small fractions of plagioclase, potassium feldspar and hematite. The firing temperature of this ceramics has not yet been determined.

#### Discussion and conclusion

On the basis of the microscopic, X-ray diffraction and chemical examinations it was established that a portion of the antique ceramics found in the »Ancient street« in *Salona* was made of a clayey raw material found in the flysch sediments of Middle Eocene age in the immediate surrounding of *Salona*.

The stratigraphic affiliation of the clayey material used in the production of the ceramics was confirmed by the microscopic determination of the relatively well preserved foraminifers of the ceramics. Calcite skeletons of the foraminifers underwent the preparation and processing of the clayey raw material, as well as firing ceramic products. During the firing process calcite of the microorganic skeletons turned into CaO. Calcium oxide exposed to moisture transformed into Ca(OH)<sub>2</sub>, and the latter, being in contact with carbon dioxide, transformed back to the stable calcium carbonate, calcite. The forms of the foraminifer skeletons remained almost unchanged, having undergone only slight distortions, as observed by means of optical microscopy.

Therefore, it was concluded according to the present mineralogical study of the antique ceramics and the Eocene flysch from Duilovo, that the raw clay material for preparation of the ceramics was taken in an immediate surrounding of Salona. The ratios of the oxide fractions in the ceramics and flysch were very similar. The mineral composition of the fired composite material was very similar to that of the antique ceramics RZH-2146. The ceramics originally contained illite (not destroyed during preparation) and relatively poorly defined gehlenite and diopside. Therefore, one could conclude from the comparison of the diffraction patterns of the fired ceramics and of the composite material, that the firing temperature of the antique ceramics RZH-2146 was probably between 800 and 850°C.

Besides the ceramics described above,' one more ceramic sample was analysed by X-ray powder diffraction. This contained an amount of quartz similar to that in samples RZH-2146 and RZH-2147, but it contained bigger amounts of plagioclase, potassium feldspar, hematite, muscovite and an unidentified mineral. The ceramics did not contain calcite. It was obvious that the raw clay material, used in preparation of that ceramics, was different from that used in the ceramics RZH-2146 and RZH-2147, that is, it did not originate from the Eocene flysch, found in the surrounding of *Salona*. Further examinations are needed to establish the origin and other properties of that ceramics.

#### Acknovdedgments

We are grateful to the Croatian Restoration Institute, Zagreb, for the permission to publish the present work. We would also like to thank the teams of the Croatian Restoration Institute and the Archaeological Museum, Split, who carried out the field work on the site by collecting the samples. Our thanks are also due to Dr D. Šiftar who carried out the chemical analysis of the ceramics and flysch.

## Reference

Malinar, H. and Smailagić, Lj. (1987): Elaborate - Conservatory protection of archaeological objects of ancient Salona in sectors »Ancient street« and »Hortus Metrodori« (in Croatian: Elaborat - Zaštita arheoloških iskopina antičke Salone na sektorima »Antička ulica« i »Hortus Metrodori«), Croatian Restoration Institute, Zagreb.

	ceramics RZH-2146	flysch, insol. residue
SiO,	44.35	56.98
$A1_{2}O_{3} + TiO_{2}$	12.53	16.20
FeO	n.d.	0.87
Fe <sub>2</sub> O <sub>3</sub>	4.96	6.57
MgO	1.92	2.58
CaO	18.07	1.70
Na,O	0.90	0.70
K,Ô	1.52	2.64
H <sub>2</sub> O (105°C)	1.87	4.85
Ign.loss(105°-600°C)	4.30	4.94
Ign.loss(600°-900°C)	9.46	1.31
	99.88	99.34

Table 1. The chemical composition of the ceramics and the flysch insoluble residue (weight percent)

Table 2. The proportions of the oxides in the ceramics and the flysch

oxides	ceramics RZH-2146	flysch, insol. residue
$A1_2O_3$ :SiO <sub>2</sub>	0.283	0.284
$Fe_2O_3$ : SiO <sub>2</sub>	0.112	0.133
$MgO : SiO_2$	0.043	0.045
$K_2O$ :SiO <sub>2</sub>	0.034	0.046
MgO : $A1_2O_3$	0.153	0.159

## FIGURE CAPTIONS

Fig. 1.

Position map of *Salona* near Split, with the newly discovered Roman sites »Ancient street« (A) and »Hortus Metrodori« necropolis (B) and position of Duilovo east of Split.

Fig. 2.

Microphotographs of ceramic sample RZH-2146, shovving well preserved calcite skeletons of foraminifers and particles of quartz and quartzite in the cryptocrystalline microscopically indeterminable matrix. Cross Nicols.

Fig. 3.

Microphotographs of ceramic sample RZH-2147, showing not well preserved and partly distorted calcite skeletons of foraminifers and particles of quartz and quartzite in the microscopically indeterminable matrix. Cross Nicols.

Fig. 4.

X-ray powder diffraction patterns of ceramic sample RZH-2146 (a), and of the same sample after treatment by 1.5% acetic acid (b).

Fig. 5.

X-ray powder diffraction patterns of ceramic sample RZH-2146 fired for 3 hours at 750°C (a), 800°C (b) asnd 900°C (c).

Fig. 6.

X-ray powder diffraction patterns of ceramic sample RZH-2146 (a) and of the composite sample (b), both fired at 900°C for 3 hours.

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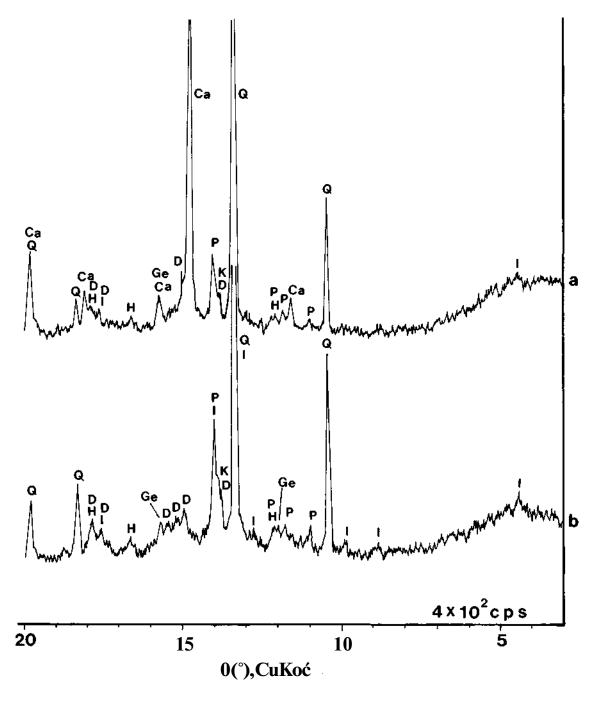


Figure 4

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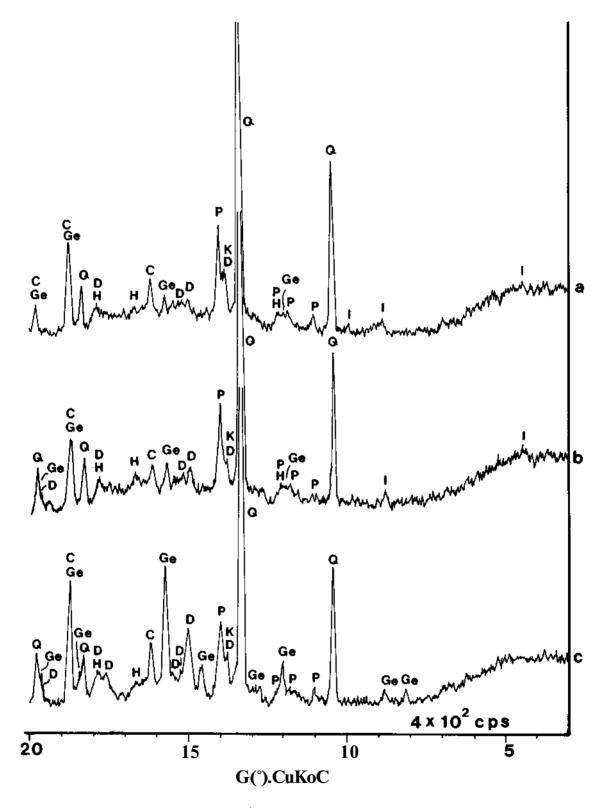


Figure 5

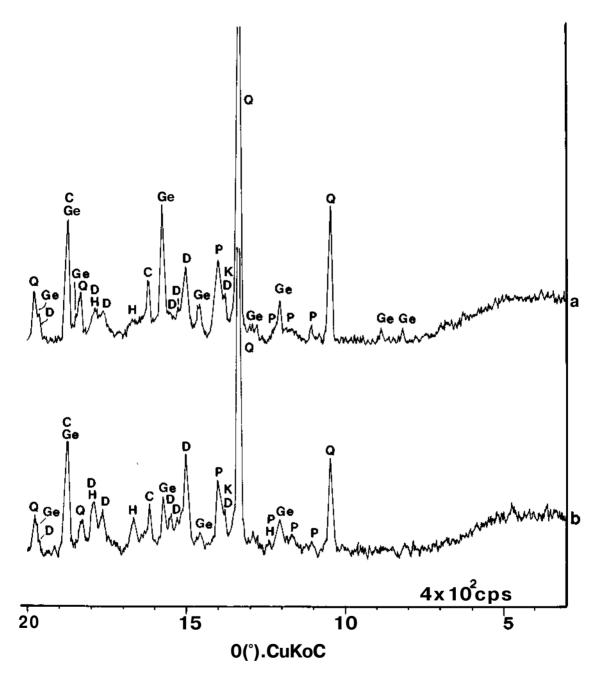


Figure 6