

Petrology and geochemistry (fluid inclusions) of Miocene halite rock salts (Badenian, Poland)

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Abstract. Within Badenian evaporites of southern Poland, three sections have been selected for petrological and geochemical study. Rock salt samples from the vicinity of Wieliczka and Upper Silesia were studied in thin sections under microscope and examined by XRD analysis. A number of selected samples were tested by fluid inclusions microanalysis using the Cryo-SEM-EDS methodology. As a result of this study, the mineralogical components and characteristic textures of the rock salt have been established. Fine to coarse crystalline halite is the main constituent of the rock salt. Relics of large hopper crystals are abundant in two of the studied sections. Anhydrite and gypsum are the occurring sulphate minerals, the latter being derived mainly from partial anhydrite hydration. Polyhalite is absent or very scarce. The chemical composition of primary fluid inclusions is in accordance with a marine origin of the deposit. The homogeneity of the fluid inclusion composition in the analyzed samples suggests that the mother brines reached a steady state during the halite deposition. To reach this steady state, Na and Cl lost by halite precipitation had to be added by inflow waters enriched in both elements so that a recycling mechanism was needed. With the available data it is not possible to determine the source (marine, continental or mixed waters) of the inflow implicated in the recycling mechanism.

Key words: Badenian salts, evaporite petrology, halite, fluid inclusions composition, Poland.

Introduction

Halite is the most frequent mineral precipitated during the evaporation of marine, continental, mixed, or hydrothermal waters. However, the solute composition of these saline brines undergoes important variations that are not recorded in the mineral precipitation. The direct analysis of the electrolyte composition of primary fluid inclusions in the halite crystals of ancient evaporite formations is the best method to identify the evaporation stage attained by the original brines.

The aim of the present paper is to characterize the chemical composition and origin of the mother brines of the Badenian halite rock salts of Poland by means of the analysis of fluid inclusions in primary halite crystals. Prior to fluid inclusion study, the mineralogical composition and textures of the halite salt rocks were established in order to recognize the sedimentary and diagenetic features of the deposit.

Geological setting

The Badenian Stage (M₄, Middle Miocene) of the Carpathian Foredeep in Poland has been subdivided into a

number of lithostratigraphic units. The oldest unit is known as the Skawina Beds (Fm) and consists of marly mudstones, siltstones, and shales. These sediments are up to 500 m thick in Upper Silesia and from 600 to 800 m thick in the area east of Kraków, Poland (Fig. 1).

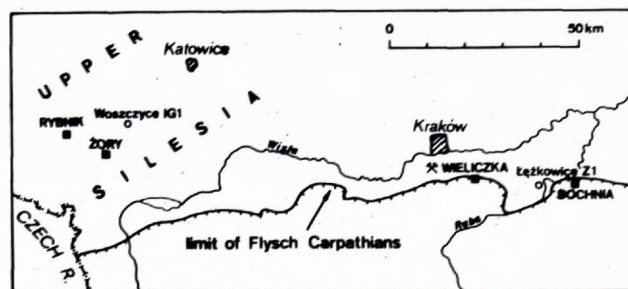


Figure 1 Location of profiles in Upper Silesia and in vicinity of Wieliczka salt deposit.

The Wieliczka Beds (Fm) overlie the Skawina Beds and are composed predominantly of evaporites (Garlicki, 1994a). In the southern part of the Badenian basin (Upper Silesia and the area east of Kraków) the Wieliczka Beds consist of rock salt and anhydrite. The normal thickness of

these sediments does not exceed 100 m. The Chodenice Beds overlie the Wieliczka Beds and were deposited during the final stage of an evaporite basin, in an euxinic environment. These sediments are therefore dark, thinly laminated, and contain a considerable amount of chlorides, sulphates, and carbonates.

Within the Badenian evaporites of southern Poland, several salt sections in the area of Upper Silesia and vicinity of Wieliczka have been recognized and correlated (Garlicki, 1971, 1974, 1993). Besides the Wieliczka salt deposit, among these sections, Woszczyce IG-1 and Łęzkowice Z-1 have given recently new materials for core examination and sampling (Fig. 1). However, the rock salt beds at the Wieliczka salt deposit are of utmost significance (Garlicki & Wiewiórka, 1983).

Sedimentary cyclothems and marker beds distinguished in the bedded part of the Wieliczka salt mine made it possible to carry out a lithostratigraphic correlation in a regional scale (Garlicki, 1979, 1994b). It has been stated that salts of cyclothem I were deposited only in the area between Bochnia and Tarnów. In cycle II, the sedimentation of salt covered an area from Upper Silesia to Tarnów. During that period, the oldest salts (N) of Wieliczka were deposited (Fig. 2). In cyclothem III the sedimentation of salt had the greatest extent, ranging from Upper Silesia to the area east of Tarnów. At Wieliczka, these sediments contain the following marker beds: ZP - green layered salts, SZ - shaft salt, SD - lower spiza salts. Corresponding similar sediments are found in neighbouring sections (Fig. 2). The cyclothem IV includes in Wieliczka area the upper spiza salt (SG) similar to the lower.

Results and interpretation

A total number of 25 samples were selected corresponding to three salt sections: the borehole Woszczyce IG-1 in the west (Upper Silesia), the Wieliczka salt mine in Kraków area, and the borehole Łęzkowice Z-1 in the east (Bochnia area) (Table 1, Fig. 1, 2).

The halite samples were collected in the autochthonous parts of the sections. In the Wieliczka section, additional samples numbered 6 and 7 were collected from the uppermost brecciated part of the deposit (not shown in Fig. 2), which primarily was deposited in the southern zone of the sedimentary basin. This upper unit consists of a coarse breccia composed mainly of salt clays with irregular blocks of coarse grained salt. The Chapel of Blessed Kinga (54 m long, 14.5 m wide, and 10 m high) is located in one of the largest blocks.

Petrology

In the studied samples halite is the main constituent. It is fine (150 µm) to coarse crystalline (up to 3 cm). Relics of randomly oriented hopper crystals are abundant in

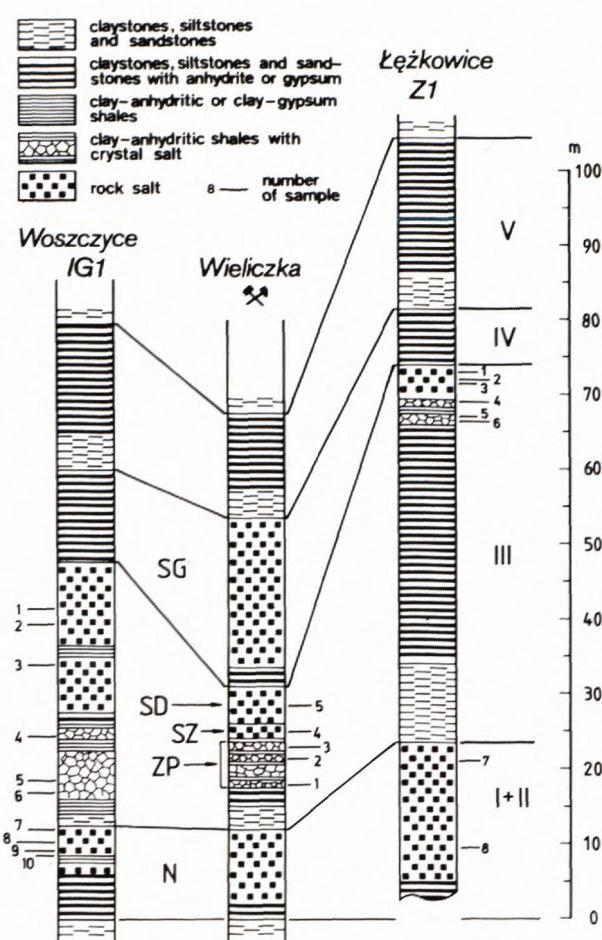


Figure 2 Lithostratigraphic correlation of salt sections Woszczyce IG-1, Wieliczka, and Łęzkowice Z-1.

samples from Woszczyce IG-1 and Łęzkowice Z-1, and scarce in samples from the autochthonous bedded deposit of Wieliczka. Neither chevron nor cornet structures were observed, although the former have been reported from Wieliczka salt mine in the literature (Czapowski, 1994).

Anhydrite and gypsum are the occurring sulphate minerals, the latter being derived from partial anhydrite rehydration. Polyhalite was neither detected by XRD nor clearly identified under microscope. Anhydrite shows different varieties: microcrystalline nodules (up to 1 cm), individual laths (up to 0.8 mm) or rectangular crystals (up to 2.4 mm) and poikilitic cements in the muddy component. Tiny euhedral gypsum crystals were observed inside some fluid inclusions; we interpret them as primary trapped crystals so that the original paragenesis was halite-gypsum (García-Veigas *et al.*, 1991).

Dolomite is scarce, but equant crystals, from 30 to 120 µm, were identified by SEM in samples from Woszczyce IG-1.

The fine-grained detrital component is scattered in general, but can occur as thin laminae up to 1.5 cm thick in

Table 1 Sampling and studies carried out in Badenian halite rocks from Poland. Facies: N - oldest salts, ZP - green salt, SZ - shaft salt, SD - lower spiza salt. Mineralogy (abundant minerals are indicated in capital letters): h - halite, g - gypsum, a - anhydrite, q - quartz, d - dolomite, c - celestite.

| | Depth (m.) | Facies | Mineralogy (XRD) | Thin section study | Fluid inclusion study | |
|-------------------|---------------|---------|---------------------|--------------------------|-----------------------------|---|
| WIELICZKA MINE | | | | | | |
| | 1 | ZP | H-a-q-g | | | |
| | 2 | ZP | H-a-g | + | | |
| | 3 | ZP | H-G-a-q | + | | |
| | 4 | SZ | H-a | + | | |
| | 5 | SD | A-a-g | + | | |
| | 6 | Breccia | H-a-g | + | | |
| | 7 | Breccia | H-q-g | + | | |
| ŁĘŻKOWICE Z-1 | | | | | | |
| | 1 | 77.0 | SD-SZ | H-a | + | + |
| | 2 | 78.0 | SD-SZ | H-a | + | |
| | 3 | 78.5 | SD-SZ | H-a | + | |
| | 4 | 80.9 | ZP | H-g-a | + | |
| | 5 | 83.1 | ZP | H-g-a | + | |
| | 6 | 83.5 | ZP | H-g-a-q | + | |
| | 7 | 129.3 | N | H | | + |
| | 8 | 140.7 | N | H-a | + | + |
| WOSZCZYCE IG-1 | | | | | | |
| | 1 | 291.5 | SD | H-a-g-c? | + | |
| | 2 | 293.3 | SD | | + | + |
| | 3 | 298.6 | SZ | H-g-a-q | + | |
| | 4 | 308.8 | ZP | H-a-g | | + |
| | 5 | 314.4 | ZP | H-a-g | | |
| | 6 | 315.2 | ZP | H-g-a-d-q | + | |
| | 7 | 320.5 | N | H-g-a | + | + |
| | 8 | 322.8 | N | H-a-g | + | |
| | 9 | 324.2 | N | H-a-g | + | |
| | 10 | 324.7 | N | H-g-a | + | |

one sample (Łęzkowice, sample 5). It is formed by carbonate minerals and clay. The former were not clearly detected by XRD except in one sample (dolomite in Wieliczka, sample 6). However, under the microscope carbonate micrite can be identified and by SEM observation calcite, and dolomite pellets were detected in some samples from Woszczyce IG-1. Opaque minerals (pyrite) are also frequent. In the Wieliczka samples muddy component contains abundant quartz grains and rock fragments.

1. Salt Mine Wieliczka

In green layered salts (ZP), halite is coarsely crystalline, with scattered fluid inclusions. It contains anhydrite as laths (from 25 to 150 μm long) and light blue nodules approximately 1 cm in diameter; they are partly hydrated to secondary gypsum. The fine-grained detrital component

is also present and contains angular quartz grains (15 to 100 μm) and rock fragments (shales, phyllites) in a clayey matrix, resembling the texture of wackes. Glauconite and hematite also occur.

In shaft salt (SZ) and lower spiza salt (SD), halite is medium to coarsely crystalline (from 200 μm to 1 cm). Crystals are anhedral and some of them are elongated. Primary fluid inclusions are scarce and only secondary fluid inclusions, caused by grain boundary migration, can be observed. Anhydrite occurs in the halite grain boundaries, inside halite crystals as prismatic/rectangular crystals (up to 1.2 mm long), and also occurs in microcrystalline (fibrous?) zones, with larger crystals around them. Some anhydrite crystals are partly hydrated to secondary gypsum. The fine-grained detrital component is scarce and contains mostly quartz grains and rock fragments.

In the brecciated deposit, one of the studied samples contains coarsely crystalline halite (1 cm across). In fine-grained detrital zones smaller euhedral halite crystals occur. Primary fluid inclusions are abundant in relics of hopper crystals. Anhydrite occurs in the halite grain boundaries and also inside the halite crystals as scattered crystals (0.4 mm long), and as rounded zones (nodules?) (1.3 mm across). Anhydrite is partly hydrated to secondary gypsum. The fine-grained detrital component is present in the halite grain boundaries and also constitutes a discontinuous lamina. The second sample studied is a large halite crystal (4 cm long) with tiny primary fluid inclusions (hopper crystal).

2. Łęzkowice Z-1

In the oldest salts (N), halite is medium-to-finely crystalline. Smaller euhedral crystals (250 μm - 0.5 mm) occur associated with the fine-grained detrital component. Larger anhedral crystals are elongated and up to 8 mm long. Primary fluid inclusions are frequent in the larger halite crystals being relics of hopper crystals. Zones of clear halite are also present. Secondary fluid inclusions originated by grain boundary migration can be observed. Anhydrite forms nodules (3.5 mm across) with a microcrystalline texture. Larger prismatic anhydrite crystals arise from the periphery of these nodules. Anhydrite is also present as scattered laths (up to 0.8 mm long). These laths are absent in clear halite zones. Fine-grained detrital component occurs among the halite grain boundaries and is also lacking in clear halite zones.

In green layered salts (ZP), halite is coarse crystalline (1.5 cm) but euhedral crystals (200 μm - 1 mm) occur associated with the fine-grained detrital horizons. Primary fluid inclusions can be observed in relics of hopper crystals (8 mm - > 2 cm). Clear halite zones are also present. Secondary fluid inclusions originated by grain boundary migration also exist. Anhydrite occurs in two varieties: nodules replaced by secondary gypsum porphyroblasts

preserving anhydrite relics, and tiny anhydrite laths (80 μm long). The fine-grained detrital component occurs around the anhydrite nodules and among the halite crystals, and can also form laminae. Sample 6 is petrographically similar to the oldest salts described above.

In shaft salt (SZ) and lower spiza salt (SD), halite is medium to coarsely crystalline (up to 1 cm across). Zones of cloudy halite can be observed. Small anhydrite nodules (1–2 mm across) and scattered fine-grained detritus also occur.

3. Woszczyce IG-1 (Upper Silesia)

In the oldest salts (N), halite is mainly coarse crystalline (1 cm across) but smaller euhedral crystals (from 150 μm to 0.5 mm) are also recorded. Primary fluid inclusions are abundant corresponding to relics of hopper crystals (up to 9 mm) and secondary fluid inclusions originated by grain boundary migration also occur. Anhydrite is present in two varieties: blue coloured nodules (up to 5 mm across), often hydrated to secondary gypsum, in which larger prismatic crystals arise from the periphery of the nodules; and large rectangular crystals (up to 2.4 mm long) partly hydrated to secondary gypsum. Dolomite occurs as square ("sucrosic") crystals (from 30 to 120 μm long) that occur preferentially in the halite grain boundaries and locally are associated with fluid inclusions caused by grain boundary migration. A fine-grained detrital component is present among the halite crystals and around the anhydrite nodules. It contains tiny anhydrite crystals and opaque minerals.

In green layered salts (ZP), halite is coarsely crystalline (up to 3 cm) but smaller (less than 3 mm) euhedral crystals also exist, partly replaced by anhydrite. In some crystals primary fluid inclusions are preserved corresponding to relics of hopper crystals up to 1 cm. Anhydrite occurs as large elongated crystals associated with the fine-grained detrital component. Many of them are partly hydrated to secondary gypsum. Locally, lutecite was observed replacing anhydrite. Anhydrite also occurs as poikilitic cement in the fine-grained detrital component. This component is abundant and contains dark elongated dolomite pellets, identified by SEM-EDS, and gypsum crystals. Samples 4 and 5 contain coarse crystalline halite with relics of hopper crystals and a scattered fine-grained detrital component.

In shaft salt (SZ) and lower spiza salt (SD), halite is coarse crystalline with relics of hopper crystals and a fine-grained detrital component in variable amounts.

Bromine

All the analyzed halite samples have bromine contents between 20 and 40 ppm, in accordance with previously published data (Garlicki & Wiewiórka, 1983). These low values result from precipitation in a weakly concentrated

brine where halite recycling in slightly concentrated seawater took place (Holser *et al.*, 1972; García-Veigas *et al.*, 1995).

Fluid inclusions

The analysis of fluid inclusions was performed by the Cryo-SEM-EDS methodology described by Ayora & Fontarnau (1990) and improved by Ayora *et al.* (1994a,b) and García-Veigas (1995). This method allows quantitative analysis of the electrolytes present in fluid inclusions by direct microanalysis of frozen salt samples in SEM-EDS equipment.

In this method, a slice of halite about 1 cm^2 by 1 mm thick is placed in a sample holder with four droplets of solutions of known composition. The sample holder is then immersed in liquid N_2 and introduced in a lateral cryo-chamber of a Scanning Electron Microscope at -170°C . Inside the cryo-chamber, the mineral is broken and a flat, pristine surface is obtained, which contains a number of frozen fluid inclusions. The surfaces of the halite and the droplets are coated with aluminium in order to avoid surface electric charging. The holder is placed in the chamber of the SEM and the electron probe is focused in a few microns volume over frozen fluid inclusions or droplets and an Energy Dispersive Spectrum (EDS) in a Si-Li detector is obtained. The work conditions are established at 15 kV of probe energy, 1.5 nA of beam current and 200 s counting time. The peak-to-background ratio (I_p/I_b) of the EDS peaks for the elements present in the droplets defines a linear regression where the (I_p/I_b) of the elements in fluid inclusions are interpolated, to obtain the chemical composition of the fluid inclusions. This methodology allows the quantitative analysis of major solute chemistry (Na, Mg, K, Ca, Cl and SO_4) in natural brines trapped in primary fluid inclusions in halite.

Due to the scarcity of primary fluid inclusions in the halite from the Wieliczka salt mine, the determinations were performed in samples from the boreholes Łęzkowice Z-1 (Bochnia area) numbered 1, 7 and 8, and Woszczyce IG-1 (Upper Silesia) numbered 2, 4 and 7. The results of Cryo-SEM-EDS analysis are shown in Table 2.

The composition of the fluid inclusions corresponds to the system Na-Cl-Mg- SO_4 -K that can be considered as a marine brine. The average compositions (Table 2) are similar to the composition of concentrated seawater at the beginning of halite precipitation (McCaffrey *et al.*, 1987) (Table 3).

All the analyzed fluid inclusions have very similar compositions suggesting that the mother brines did not vary significantly from Upper Silesia to the Bochnia area. This is in agreement with the idea that the three sub-basins described in the Badenian evaporite basin had free and continuous water/brine exchange (Garlicki, 1979). Differ-

ences may be caused by minor variations in the composition of the mother brines, or due to analytical errors of the technique (less than 10%).

On the other hand, the homogeneity in the fluid inclusion compositions, from the oldest salts to the lower spiza salts suggests that the original brines reached a steady state during the deposition of the halite recorded in the studied sections. This steady state can be attained if the amounts of Na and Cl lost by halite precipitation are compensated by the inflow of a brine enriched in both elements so that a recycling mechanism of an older halite deposit is necessary.

Table 2 Average composition (in mol/l) of the components in the fluid inclusions analyzed from the Badenian salt deposit in Poland. Analyses performed by Cryo-SEM-EDS

| | | Na | Mg | SO ₄ | Cl | K |
|-----------|---|------|------|-----------------|------|------|
| ŁĘŻKOWICE | | | | | | |
| Z-1 | | | | | | |
| | 1 | 4.27 | 0.54 | 0.14 | 5.46 | 0.15 |
| | 7 | 4.39 | 0.50 | 0.12 | 5.50 | 0.06 |
| | 8 | 4.28 | 0.44 | 0.12 | 5.52 | 0.07 |
| WOSZCZYCE | | | | | | |
| IG1 | | | | | | |
| | 2 | 4.14 | 0.57 | 0.15 | 5.52 | 0.13 |
| | 4 | 4.38 | 0.57 | 0.14 | 5.57 | 0.08 |
| | 7 | 4.30 | 0.44 | 0.14 | 5.46 | 0.07 |

Table 3 Composition of Inagua brines during the evaporation path of seawater at several stages: inflow seawater, beginning of gypsum precipitation, beginning of halite precipitation, first brine evaporated in laboratory and beginning of potassium and magnesium salts precipitation. Original data from McCaffrey *et al.* (1987).

| | Na | Mg | SO ₄ | Cl | K |
|-----------------|------|------|-----------------|------|------|
| Seawater | 0.49 | 0.05 | 0.03 | 0.59 | 0.01 |
| Gypsum | 2.05 | 0.23 | 0.12 | 2.44 | 0.04 |
| Halite | 4.48 | 0.51 | 0.118 | 5.24 | 0.10 |
| Laboratory | 2.52 | 1.93 | 0.69 | 5.44 | 0.36 |
| Potassium salts | 0.63 | 3.53 | 1.19 | 5.91 | 0.67 |

Discussion

The geochemical study (fluid inclusion composition and bromine contents in halite) of Badenian rock salts of the Carpathian Foredeep in Poland indicates that halite is partly recycled in origin. The recycling of early halite deposits located at the basin margin is a common process at the final stage of many saline formations, as it has been reported in the Messinian salt deposits of Central Sicily basin and Lorca basin (Spain) (García-Veigas *et al.*, 1995) and in the Eocene potash basin of Navarra (Spain) (Ayora *et al.*, 1994b).

As stated above, the homogeneity in the composition of fluid inclusions (steady state) and the low bromine

contents in salt, can be achieved by the inflow of waters enriched in Na and Cl by dissolution of an older halite deposit. With the available data it is not possible to determine the source of the inflow (marine, continental or mixed) or the evaporitic formation recycled (the Zechstein salt rocks or the earlier halite deposits of the Badenian basin).

In order to maintain the halite precipitation in a steady-state condition, an inflow enriched only in Na-Cl is required but, in this case, the contents of potassium, magnesium, and sulphate would be lower than those obtained in the analyzed fluid inclusions because the mother brine would achieve the saturation in halite in an early stage of concentration. This situation is recorded in the composition of fluid inclusions in the upper section of the Messinian halite formation of Lorca basin (Spain) (Ayora *et al.* 1994a; García-Veigas, 1995) where the brine composition shows a continuous decrease in Mg and K, and an increase in Na contents.

Although the composition of the fluid inclusions studied is very similar to the composition of seawater at the beginning of halite precipitation, it could also be achieved by the evaporation of a special type of continental waters (Ayora *et al.*, 1994a). However, the connection of the Badenian Carpathian Foredeep with the open sea is accepted in the literature (Rögl & Müller, 1978; Czapowski, 1994).

The relics of hopper halite crystals that exist in the studied halite samples show some features that are in accordance with the idea that halite precipitation took place in a calm brine body that had reached a steady state composition. These crystals have an inner core where fluid inclusions are disorientated suggesting the equilibrium of the brine, without a marked salinity gradient, and with no variations and no supersaturation. This situation can be achieved in a deep brine or in a stratified brine. The orientation of fluid inclusions that can be observed in the outer part of these crystals is related to their large size: when halite crystals reach a critical size, a preferential development of cube edges over cube faces occur due to a mass transfer problem (Batchelder & Vaughan, 1967).

Polyhalite is absent or, at least, very scarce in Badenian salt deposit. This mineral grows interstitially during early diagenesis replacing initially a precursor sulphate mineral (gypsum or anhydrite) and after the halite crystals. The halite replacement starts from the grain boundaries surrounded by muddy sediment. Primary sedimentary deposits of polyhalite are unknown, as far as we know. In the predicted evaporation sequences this mineral precipitates only if potassium is abundant in the brine, and also magnesium, although in a lower degree. In the Eocene potash formation of the Navarra basin polyhalite occurs in the lower part, below the potash beds, but is lacking in the upper halite unit which formed from a K-poor brine

(Rosell, 1983; Ayora *et al.*, 1994b). In the halite samples studied from the Badenian salts, potassium contents are also very low in the fluid inclusions indicating a low concentration of this element in the mother brines.

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