FACIES ANALYSIS AND FLUID INCLUSION STUDIES OF THE MESSINIAN EVAPORITES, CALABRIA, SOUTHERN ITALY

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INTRODUCTION

The Messinian salinity crisis (MSC), occurred around 6 Ma ago, is a complex geological event which transformed the Mediterranean Sea into a giant saline basin causing a catastrophic hydrological and biological crisis (CIESM, 2008). The peculiar geological event triggered the formation of extensive evaporite deposits which, in less than 640 ka, accumulated on the bottom of the Mediterranean basin (Roveri *et al.*, 2008; Lugli *et al.*, 2010; Manzi *et al.*, 2009, 2012) known as Lower Evaporites, Halite and Upper Evaporites. Over the years, numerous scientific studies have been carried out on several deposits dislocated in the Mediterranean basin (*e.g.*, Spain, Central-Northern Italy, Sicily, Turkey, Cyprus) contributing to better understanding of their formation mechanisms. However, only stratigraphic and petrographic preliminary study (Barone *et al.*, 2007; Lugli *et al.*, 2007; Cianflone *& Dominici*, 2011; Cianflone *et al.*, 2012; Speranza *et al.*, 2013; Costanzo *et al.*, 2019) have been conducted on the evaporite deposits located in the Calabria region (Southern Italy), leaving many unresolved questions. In detail, in the Calabria region there are different evaporite outcrops associated with the main MSC deposition events: halite salt domes in the Crotone basin and selenite and gypsum deposits in the Catanzaro and Ionian Basin.

The focus of this work is to increase the knowledge on the geological evolution of the Calabrian basins during the Messinian age through a multidisciplinary approach (petrographic, fluid inclusion and geochemical studies), placing the Calabrian evaporite into the interpretive framework of the Messinian salinity crisis for the first time. The main aims of this project are: *i*) the identification and description of the a) different halite and selenite facies (primary or secondary), b) biological content, c) deformation degree of the crystals (plastic/ductile strain); *ii*) reconstruction of the paleoenvironmental and paleoclimatic conditions (salinity, temperature of homogenization, chemical composition); *iii*) dating the crystals; *iv*) interpretation of the sedimentological facies and their relationships with the hydrological and geochemical factors characterizing the original depositional environments: *v*) creation of new depositional models for the halite and selenite outcrops.

GEOLOGICAL SETTING

The Crotone basin, the Catanzaro Trough and the Ionian Basin are three Neogene-Quaternary basins respectively developed in the North East, central and South East of the Calabrian Arc (Fig. 1).

The Crotone Basin is generally interpreted as a forearc basin positioned on the internal part of the Calabrian accretionary wedge (Bonardi *et al.*, 2001; Barone *et al.*, 2008; Zecchin *et al.*, 2013). During the Miocene, the basin registered the evaporite deposition consisting of clays saliferous, gypsum and halite. We investigated salt domes outcrops in the Coste del Sale, Zinga and Verzino areas.

The Catanzaro Trough is a graben located between the Serre and the Sila Massifs, and filled by up to 2000 m of continental to marine deposits. A small part of this infilling (about 50 m) includes selenite deposits formed during the Messinian age (Cianflone & Dominici, 2011, Costanzo *et al.*, 2019) and cropping out in the Marcellinara area (disused quarry).

The Ionian Basin is located between the Aspromonte and Serre Massifs and is characterized by sedimentary successions, Cretaceous to Lower Miocene allochthonous terranes, covered by Oligocene to Quaternary deposits (Dominici & Sonnino, 1999, Critelli *et al.*, 2016). The evaporite deposition consist of limestone (Calcare di Base



Fig. 1 - Lithological maps of miocenic and pliocenic sediments and points sampled in the Calabria region. Halite crystals sampled from salt domes located in the Coste del sale, Zinga and Verzino (Crotone Basin), selenite/gypsum crystals from the Marcellinara quarry (Catanzaro Trough) and Benestare quarry (Ionian Basin).

Formation), massive or laminated, vacuolar, brecciated, microcrystalline limestone, selenite deposit and gypsarenite. The selenite deposit investigated is located in the Benestare area (disused quarry).

METHODS

Sixty-one samples from exposed outcrops were analysed: *i*) twenty halite crystals from the Coste del Sale, Verzino and Zinga; *ii*) twenty-five selenite/gypsum samples from the Marcellinara quarry; *iii*) sixteen selenite samples from the Benestare quarry. The most suitable samples were sent to the ALS Petrophysics laboratory (Normandy), where 22 wafers of about 500 µm in thickness for the halite and of about 200 µm for the selenite/gypsum were prepared.

The petrographic investigations were carried out using the Optical Microscopy, Ultra-Violet Microspectroscopy, Laser Raman Microspectroscopy and Scanning Electron Microscope. The chemical parameters to obtain information on the paleoenvironmental and paleoclimatic conditions (salinity, temperature of homogenization, brine composition) were carried out through microthermometry using the Linkam THMG-600 heating-freezing stage mounted on a Nikon Labophot transmitted light microscope. The dating of the crystals for the accurately placing in the MSC scenario were carried out using ⁸⁷Sr/⁸⁶Sr ratio measurements. This procedure was carried out in an ISO 6 class clean room at the mass spectrometry laboratory at Department of Earth, Environment and Resources - University of Naples Federico II.

The overall analyses were conducted either in the Geofluids Research Laboratory at the NUI Galway (Ireland) or at the University of Calabria (Italy).

PETROGRAPHIC OBSERVATIONS

Halite salt domes from the Crotone Basin

The Crotone Basin records three facies: banded, white and transparent (Fig. 2).

Banded facies (microcrystalline halite) consists in repetition of centimeter to decimeter thick dark and white layers made of halite. Crystals show elongate and rounded shapes up to 5 mm in length and competitive growth that determines mosaic texture. Clay minerals and halite cement are dispersed throughout the samples.

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Fluid inclusions are not visible to the naked eye. This facies was recognised in the Coste del Sale, Zinga and Verzino areas.

White facies (macrocrystalline halite) is made of massive clear halite crystals (> 5 cm in size). Crystals show milky (enriched in fluid inclusions) and white (impoverished in fluid inclusions) bands visible to the naked eye. Alternating bands define primary structures called as "chevrons shape" that indicates edges growth (Warren, 1999, 2006). Sometimes the crystals show sharp and curved dissolution surface along the edges. Rare evidence of dissolution and recrystallization processes (fractures) which have not obliterated the primary structures, allowed us to suppose that the deposit is not recrystallized.

<u>Transparent facies (macrocrystalline halite)</u>, so called for its aspect, is made of massive and transparent crystals (> 5 cm in size). No preferential habits have been observed. Fluid inclusions throughout the sample are visible to the naked eye. This facies has not been discussed in previous literature.

The white and transparent facies were recognised in the Zinga area in the same deposit (in continuity of deposition).



Fig. 2 - Halite samples from the Crotone basin. A) microcrystalline halite belonging to the banded facies; B) macrocrystalline halite from the white facies; C) macrocrystalline halite from the transparent facies.

Selenite crystals from the Marcellinara quarry

The Marcellinara quarry was divided in six sectors during the sampling and records three facies (Fig. 3): *giant* - documented as individual blocks (sector 1), *banded* - recognised in two deposits (sector 4 and 5), and



Fig. 3 - Selenite/gypsum samples from the Catanzaro Trough. A) Giant selenite; B) selenite crystals belonging to the banded facies and C) gypsum samples belonging to the branching facies from the Riato conglomerates.

branching – identified as blocks in the Riato Conglomerate (close the quarry). The other sectors show chaotic deposits made of reworked massive selenite. The *giant facies* shows twinned selenite crystals with vertical growth. The blocks are larger than 1 meter.

The *banded facies* is made of twinned selenite crystals (< 10 cm in size) that form palisade structures. This facies is separated by dissolution surface and breccia layers in the sector 4 and by thin argillaceous/calcareous laminae in the sector 5.

Banded and giant facies have the same petrographic features. Microscopically, show the alternation of turbid and limpid bands visible to the naked eye (microfacies) called cloudy and clear intervals. Cloudy intervals are enriched in organic and inorganic matters; in contrast, clear intervals are depleted in these matters.

The *branching facies* shows lenticular microcrystalline gypsum crystals (size < 2-3 mm). Microscopically, shows chaotical gypsum crystals immersed in a brown-yellow-orange matrix (calcium sulphate). The splintered crystals display the twin plane, chaotic orientation and different shape (elongated, pentagonal or irregular).

Selenite crystals from the Benestare quarry

Macroscopic observations on the deposit suggested the presence of two facies: banded and branching. All samples collected show fractured and folded crystals with chaotic orientation immersed in a yellow-brown matrix. These evidences allowed us to think that this deposit is secondary in origin.

ORGANIC MATTERS

In the halite and selenite crystals, organic matter is trapped both in the crystals and in FIs (Fluid Inclusions). It displays a considerable range of colour, morphology, abundance and distribution. Under UV light, it shows strong to faint blue, red or green fluorescence. Petrographic and SEM observation highlight nine different organic matter types. These have been identified as: 1) blue microalgae (Oscillatoriales) displaying strong blue fluorescence under UV light; 2) green microalgae belonging to Chlorophytas, showing strong green fluorescence; 3) red microalgae (Rhodophyta) displaying strong red fluorescence; 4) green algae (Dunaliella cells) showing strong red fluorescence; 5) green/yellow algae (Nannochloropsis or Chroococcidiopsis) with or without green fluorescence; 6) globus geosporum spores that do not display fluorescence; 7) foraminifera emitting strong to none fluorescence; 8); biofilm structures of cyanobacteria that do not display fluorescence; 9) coccoliths and radiolarian recognised trough SEM.

In all halite samples blue/red algae and Dunaliella cells were recognised. In selenite crystals the organic matters are mainly trapped in the cloudy intervals (all varieties listed above), whereas the clear intervals trapped only blue algae and Dunaliella cells.

FLUID INCLUSIONS INVESTIGATION

Halite crystals

In halite crystals primary, secondary and pseudo-secondary FIs were observed. Primary FIs (Type 1 - monophase liquid inclusions, Type 2 - Two-phase - liquid-rich inclusions and Type 3 - multiphase solid inclusions with crystals and organic matters) run parallel to the faster growth faces (100) and show mainly tabular and negative crystal shapes. Inclusions are distributed in linear trails, in groups or as individual inclusion. We focused on the primary FIs.

The banded facies show primary FIs never observed in previous studies (see Lugli *et al.*, 2007). Primary FIs are trapped along the crystal surface imperfections (rapid growth) and sometimes can trap small air bubble (Robert & Spencer, 1995); if dissolution processes during their growth and deposition are not marked, crystals can show growth zonation (relicts primary structures) highlighted by FIs distribution. In the samples from Coste del Sale and Zinga, the relict structures are more visible than samples from Verzino which instead show fractured and deformed crystals and more re-equilibration phenomena associated to plastic (stretching) and ductile (leakage) strain during burial and/or exhumation (Vityk *et al.*, 2000) that partially removes the primary structures. The presence of Type 3 inclusions with trapped organic matters, testify a not complete recrystallization.

The white facies shows primary and rare secondary FIs. Primary fluid inclusions (already observed by Lugli et *al.*, 2007) define chevron structures (Goldstein & Reynolds, 1994) that indicate growth pulses. Each pulse is characterised either by thin layers of less saline salts or by inclusions of brine that were poikilitically

enclosed during crystal precipitation. This crystal layering reflects high-frequency of change of the precipitation rate in crystals induced by short-term changes.

The transparent facies shows trails of primary FIs and rare secondary inclusions. The trails define chevron morphologies with a lower frequency of pycnocline oscillation than white facies. Abundant FIs Type 3 (with organic matters) indicate a primary origin of this facies, confirmed by faint re-equilibration and necking down process.

Selenite crystals - Catanzaro Trough

In selenite samples from the banded facies, 1149 FIs were observed. The greatest amount of inclusions is trapped in the cloudy intervals because during their formation, the crystals were disturbed by fresh water inflow that generated crystallographic irregularities favouring the FIs entrapment; in contrast, during the clear interval's formation, the crystals grew in a stable environment, trapping occasional FIs. The observed numerous FIs shapes reflect the crystallographic disorders. In detail, the cloudy intervals show a greater number of Type 2 FIs (distributed in linear trails) than clear intervals, whereas, the clear intervals show an increase of Type 1 FIs (distributed in group o as individual FI). All crystals preserve primary features. Necking down process, re-equilibration phenomena and fractures have only partially obliterated these structures. Contrarily, in the sample belonging to the branching facies from the Riato conglomerate, primary structures were almost completely deleted.

Selenite crystals - Ionian Basin

In all selenite samples, FIs study allowed us to affirm that the crystals have undergone many deformations after their deposition because the occurring few inclusions are not easily ascribable to primary or secondary origin. Primary structures have been cancelled. These observations made us to suppose that this deposit is of secondary origin.

MICROTHERMOMETRIC ANALYSIS

Problems on the microthermometric investigation

In evaporite minerals, fluid inclusions studies may be subject to numerous analytical and interpretation errors due to the low temperature of the crystal formation which could determine metastability and re-equilibration processes. In fact, monophase fluid inclusions could be suffering from metastability whereas the use of Type 2 FIs in microthermometric studies at low temperature is usually inadvisable. In addition, another problem of the FIs in selenite minerals is the low salinity values that come out of the microthermometric analysis. These values fall in the carbonate precipitation field and rarely in the gypsum precipitation field (Costanzo *et al.*, 2019; Natalicchio *et al.*, 2014) causing disagreement in the chemistry community. In this research, we try to define, through a multidisciplinary approach, a method of investigation for the use of the fluid inclusions, mainly trapped in selenite crystals, defining which fluid inclusions can be used to obtain chemical information.

Chemical information

According to the overall distribution in primary FIs, six different salt system were recognised according to Shepherd *et al.* (1985): 1) Temperature of first melting (T_{FM}) between -60° and -50°C is close to the eutectic temperature of the H₂O-NaCl-CaCl₂ system (-55°C); 2) T_{FM} between -54.5° and -44.5°C close to the eutectic temperature of H₂O-CaCl₂ system (-49.5°C); 3) T_{FM} between -40° and -30°C is close to the eutectic temperature of H₂O-NaCl-MgCl₂ systems (-35°C) and T = -35°C is the transition T_{FM} between the iron-rich system and the magnesium-rich one; 4) T_{FM} between -38.6° and -28.6°C is close to the eutectic temperature of H₂O-MgCl₂ system (-33.6°C); 5) T_{FM} between -28.5° and -18.5°C is close to the eutectic temperature of the H₂O-NaCl-KCl system (-23.5°C); 6) T_{FM} between -21° and -19°C is close to the eutectic temperatures of following systems: *i*) H₂O-NaCl-SO₄ (T_{FM} = -21.7°C); *ii*) H₂O-NaCl-NaHCO₃ (T_{FM} = -21.8°C); *iii*) H₂O-NaCl-Na₂Cl₃ and -20.8°C).

Using the AqSo software, salinity values were obtained through several formulas based on the phases observed during the heating. In details, the formula used were: *i*) Sterner *et al.*'s formula (1988) for the H₂O-NaCl-KCl system and positive Temperature of last melting (T_{LM}) values - oversaturated system in hydrohalite (T_{FM} = between 0° and +0.1°C) or halite (T_{FM} over +0.1°C); *ii*) Hall *et al.*'s formula (1988) for the H₂O-NaCl-KCl system and negative T_{LM} values; *iii*) Oakes *et al.*'s formula (1990) for H₂O-CaCl₂ the system; *iv*) Dubois & Marignac's formula (1997) for H₂O-MgCl₂ the system; *v*) Bodnar's formula (1993) for all other systems.

One hundred and sixty-eight FIs in halite crystals and two hundred and twenty-two FIs in selenite crystals were analysed.

Halite banded facies showed T_{FM} between -45° and -25°C, salt system made up H₂O-NaCl-MgCl₂, H₂O-MgCl₂ and H₂O-FeCl₂, T_{LM} from -1.1°C to + 2.8°C, salinity values about 26.1 eq. wt.% NaCl and Temperature of homogenization (T_H) between +20°C and +40°C.

Halite white facies showed T_{FM} from -36° to -33°C, salt system made of H₂O-MgCl₂, T_{LM} from -1.8° to -1.5°C, salinity values about 26 eq. wt.% NaCl and T_{H} about +31.5°C.

Halite transparent facies showed T_{FM} from -40° to -32°C, salt system made of H₂O-MgCl₂, T_{LM} from -2° to -0.4°C, salinity values about 26.1 eq. wt.% NaCl and T_H about +21°C.

In the selenite samples, microthermometric our study revealed a great difference between cloudy and clear intervals in terms of TFM, salt system and salinity (Fig. 4).



Fig. 4 - Selenite sample with salt system composition in seven cloudy intervals (white box) and three clear intervals (pink box). On the right salinity average values for each interval. Red dotted lines indicate absence of data.

FIs in cloudy intervals show T_{FM} from -58° to -23°C, Mg, Ca, Cl as dominant elements (Na in minor amount) and T_{LM} from -4.5° to -0.2°C, (where the last solid phase is made of ice crystals) and salinity values from 0.4 to 8.2 eq. wt.% NaCl (av. about 3 eq. wt.% NaCl). These results are in agreement with those of Natalicchio *et al.* (2014), that provided low salinity with values that fit in the carbonate precipitation filed (Messinian Lower Gypsum located in North Italy - without discriminating between two microfacies).

In contrast, FIs in clear intervals show T_{FM} from -58° to -23°C, Mg, Ca, Cl as dominant elements (Na in minor amount) and T_{LM} from -4.5° to -0.2°C (where the last solid phase is made of ice crystals) and salinity values from 4.5 to 22.3 eq. wt.% NaCl (av. about 16 eq. wt.% NaCl). These evidences have never been observed so far and could testify that the crystals, during their growth, registered a middle-frequency climatic oscillation.

Fifty-eight salinity variations were documented throughout the outcrop located in the sector 5, which made us to suppose that proportion of fresh water, that fed the system, was recurring over time.

87SR/86SR ISOTOPIC STUDY

To obtain more information on the origin and time of deposition of halite, selenite, and gypsum, isotopic investigation on the strontium were conducted on all samples. This technique showed surprising results (Fig. 5) indicating that halite salt dome (⁸⁷Sr/⁸⁶Sr between 0.708918 and 0.709050) formed during the second stage of the MSC during the deposition of the Resedimented Lower Gypsum (RLG) and Halite (H) deposition step (5.60-5.55 Ma).



Fig. 5 - Sr isotope curve in the Mediterranean Sea and Global Ocean during the Messinian age. The distribution evidences the progressive change in the isotopic composition of Mediterranean waters during the Messinian salinity crisis. Figure modified after Roveri *et al.* (2014).

Banded Selenite (⁸⁷Sr/⁸⁶Sr between 0.708786 and 0.708805) formed during the third stage of the MSC during the Upper Gypsum (UP) deposition (5.43 Ma - Lago mare event - LM). Moreover, the Giant Selenite

 $(^{87}\text{Sr}/^{86}\text{Sr}$ value of 0.708797) formed in this stage and this is the first time that appear the giant facies in the third stage of the MSC, clarifying doubts about its origin.

Branching facies (87 Sr/ 86 Sr between 0.709045 and 0.709082) from the Marcellinara and Benestare quarry formed during the first stage of the MSC during the Primary Lower Gypsum (PLG) deposition (Stage 1 \rightarrow about 5.9 -5.6 Ma) and were subsequently eroded and incorporated in the Riato conglomerate during the second stage of the MSC, whereas the deposit located in the Benestare quarry was eroded and resedimented.

The isotopic analysis revels that the Calabria region is the only area around the Mediterranean that preserve facies formed in all three stage of the Messinian salinity Crisis.

DEPOSITIONAL MODELS

The halite facies from the Crotone Basin

The halite depositional model is based on the Zinga deposits because this is the only area where all three facies outcrop. In light of the new evidences and according to the "Salin Pan Cycle" of Lowenstein & Hardie (1985), the banded, white and transparent facies formed during *Evaporite concentration* in a basin with salinity of about 26.3 eq. wt.% NaCl. In detail:

a) the banded facies formed at the air-brine interface when surface water salinities were suitable. When the crystals mass became heavier, they fell to the bottom and continued to grow competitively (stables pycnocline). Crystal formation and accumulation depended on the low wind and waves intensity and stables pycnocline. Considering the clay matters recognized between crystals and the organic matters, we suppose that these facies formed in a saline environment subjected to continental inflow phenomena and mixing of the water column. For these reasons, banded facies formed during the **initial phase** of the *Evaporite concentration* in a hybrid brine with temperature between $+20^{\circ}$ and $+32^{\circ}$ C. Moreover, these samples show primary relict structures never observed in previous study on the Calabrian banded facies thus testifying a not complete recrystallization of the salt domes but different deformation due to burial, exhumation and/or active tectonic;

b) the white facies grew at the bottom of the basin and reflect changes in the crystal precipitation rate due to high pycnocline oscillation. When the water is denser (high salinity), crystal grows quickly and traps FIs; if the water is less dense (minor salinity) crystal grows slowly without trapping the FIs. This growth pulses created primary textures called "chevron fabric" that indicates fast seasonal or daily pycnocline variations. We counted 56 pulses that correspond to 28 seasonal or 28 days. This facies formed during the **intermediate phase** of the *Evaporite concentration* in a hybrid brine with temperature of about +33°C;

c) the transparent facies formed on the top of the white facies. The presence of primary FIs and organic matters testify its primary origin. A minor amount of FIs trail testify less pycnocline oscillations and a slower growth (no FIs) than white facies and formed during the **final phase** of the *Evaporite concentration* in a hybrid brine with temperature of about +21°C.

Depositional model of the Selenite deposits from the Catanzaro Trough

Starting from the bottom (deep basin) to the top (shallow basin) and considering the temporal and spatial evolution of the Marcellinara quarry outcrop, the development of the selenite deposit can thus be schematized:

1) the branching facies formed during the Lower Gypsum deposition stage (5.9-5.6 Ma) and Resedimented Lower Gypsum (around 5.6-5.5 Ma); subsequently it was eroded and incorporated in the Riato conglomerate;

2) the giant and massive facies (sector 1) belonging to the Upper Gypsum (5.43 Ma) testify the maximum brine level;

3) dome structures and reworked deposits (sector 2 and 6), which involves the previous massive facies, attest flooding events;

4) the banded facies (sector 5) formed during aridity acme, with the selenite layers indicating supersaturation condition and minimum brine level, whereas the argillaceous/calcareous laminae testify unsaturated condition.

5) the banded facies and breccia layers/dissolution surface (sector 4) mark the final stage of the aridity. Breccia layers derived from sub-aerial exposure and subsequent selenite erosion, whereas the dissolution surface testifies pycnocline variations. Deposits located in the sector 4 and 5 testify a *middle frequency climatic oscillation* between selenite layers (arid periods) and dissolution surface/breccia/argillaceous laminae (wet periods). Nevertheless, it was observed, inside the crystal, a *high-frequency climatic oscillation* between cloudy and clear intervals associated with wet and arid climate, respectively. Using the data from Schreiber & Hsü (1980) the cloudy interval (average 3 mm in size) could be formed in about 3 years, thus recording seasonal episode of *ecological bloom*.

In the light of the data obtained, we believe that the selenite banded facies was formed in a shallow basin with temperature up to $+35^{\circ}$ C (nearshore environment not exceeding 50 m of depth).

CONCLUSION

The overall objective of this research was to increase the knowledge on the Messinian Salinity Crisis, by studying the Calabrian evaporite deposits for the first time with multidisciplinary approaches.

Data obtained, prove how the Calabria is the only region, in the Mediterranean area, showing a surprising variety and diversity of evaporite rocks. In particular, this study allowed to understand that during the Messinian age: *i*) the Crotone basin had, in some areas, a geomorphological structure suitable for the formation of the different halite facies. Their formation and accumulation occurred in coastal-marine environment (no deeper than 50 m - hybrid brine) characterised by a seasonal fill; *ii*) the Catanzaro Trough was a paleochannel that received brine current from both east (Ionian Sea or eastern Mediterranean) and west (Tyrrhenian Sea or western Mediterranean). The allocation of the Giant facies at the Upper Lower Deposition is an amazing result that testify like the development of the different facies depend by the intrinsic conditions of the basin; *iii*) the gypsum deposit located in the Ionian basin is an olistostrome.

The characterization of the Calabrian Messinian deposits is the first step for the revision of the stratigraphic schemes especially for the Catanzaro Trough and the Ionian Basin, uncertain to date.

Moreover, the Calabrian evaporite deposits register a *middle-* (at the deposit scale) *and high-frequency* (at the crystal scale) *climatic oscillation* associated with wet and arid periods.

It is believed that this approach can be applied in similar areas.

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