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## Unexpected formation of sII methane hydrate in some water-in-oil emulsions

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The structures of methane hydrate obtained from emulsions of water in some crude oils have been studied. It was shown that in some of these emulsions, a hydrate of the cubic structure II (sII) instead of the expected the cubic structure I (sI) hydrate was formed when a methane-saturated sample was rapidly cooled to temperatures below  $-35\text{ }^{\circ}\text{C}$ . All experiments were carried out at a cooling rate of about  $14\text{ }^{\circ}\text{C}/\text{min}$  and a methane pressure of about 12 MPa. The sII hydrate formation in the emulsions occurred at temperatures lower than pour point of the oils. As far as we know, such an opportunity has not been previously considered in the literature. A more detailed study showed that in one of these emulsions the sII hydrate formed is metastable because sI hydrate forms in this emulsion during a long synthesis. In another case, the formation of sII hydrate can be related to both the kinetic factor (the formation of a metastable hydrate) and the presence of C3-C4 hydrocarbons in the corresponding oil in a sufficient amount. sI methane hydrate only in small amounts was formed in this oil. We assume that the reason for a metastable phase appearance in a methane –water-in-oil emulsion system is the delayed nucleation of the sI hydrate at the water –oil phase boundary. Due to this fact, the emulsions saturated with methane can be supercooled to temperatures at which the nucleation of sII hydrate is preferred.

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### Significance statement

To the best of our knowledge, this is the first time that in some methane –water-in-oil emulsion systems sII hydrates instead of sI methane hydrate can be formed. Results of this work extend comprehension of mechanisms of gas hydrate nucleation in water-in-oil emulsions at nanoscale stage.

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