

## **Acoustic Resonance Determination of the Effect of Light Hydrocarbons on Wax Appearance Points in a Njord Well Fluid<sup>1</sup>**

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Wax formation and deposition in pipelines and process equipment pose severe problems for petroleum companies, especially during transportation of crude oil in offshore environments. The light hydrocarbons present in the crude oil can play an important role in the shift of wax appearance points by increasing the solubilities of the heavier components. The following work was undertaken to study the effect of light hydrocarbons on wax appearance points in a Njord well fluid for Norsk Hydro, Norway. An automated high-pressure spherical acoustic resonator (50.8-mm-diameter) assembly designed and fabricated for that purpose has been used to measure resonance frequencies in a Njord well fluid (stabilized oil sample) provided by Norsk Hydro and blended with the appropriate amount of a synthetically prepared gaseous mixture containing six light hydrocarbons (C1 to C6), at pressures from 2 to 107 bar and temperatures in the range 35 to 50°C. Results on the present method to locate the wax appearance points in the Njord well fluid are presented. A figure showing experimental wax appearance points as a function of pressure is presented. The results are compared with those predicted by the Norsk Hydro model.

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**KEY WORDS:** acoustic resonance; cloud point; phase boundary.

### **1. INTRODUCTION**

The efficient production and processing of waxy crude oils pose serious problems, especially for operators in the North Sea region, where low temperatures promote premature dropout of heavy constituents. This unwanted behavior has prompted a number of recent efforts to construct viable mathematical models to predict the conditions under which wax

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dropout will occur in even complex hydrocarbon fluid mixtures [1-4]. As these methods necessarily require some empiricism, an appropriate experimental data bank is required for success. Although various other methods have been used to determine the occurrence of wax dropout from solution, the acoustic resonance method is especially useful because of its good sensitivity and objectivity.

The acoustic resonance behavior of a cavity bound fluid system is known to be sensitive to the system state. Abrupt changes in state, such as occur on phase transition, for example, produce corresponding abrupt changes in the acoustic resonance spectrum, and this in turn provides a basis for objective phase envelope determination [5-10]. One application of this technique is the measurement of wax appearance points in crude oil liquids. An earlier paper [8] reported on such measurements in stabilized stock tank oils. The present paper concerns an adaption of the method for the study of live fluids reconstituted in the laboratory by blending a stabilized oil with a light gas mixture under pressure. In particular, we have examined the effect of the light gas fraction on depressing the wax appearance point by increasing the solubility of the heavy (wax) fractions. The research was performed for Norsk Hydro as of Norway to provide a set of experimental data against which the predictions of an in-house model could be compared.

## 2. MEASUREMENTS

### 2.1. Materials

The stabilized oil sample, a Njord well fluid, was provided by the sponsor, Norsk Hydro a.s. (Norway). The light gas mixture was gravimetrically blended to order by Scott Speciality Gas Co., Plumsteadville, PA (USA); its composition is given in Table I.

Table I. Composition of Synthetic Gas

Component	Amount(mol %)
Methane	54.91
Ethane	17.00
Propane	12.00
<i>n</i> -Butane	9.07
<i>n</i> -Pentane	4.01
<i>n</i> -Hexane	3.01

## 2.2. Apparatus

The measurements were carried out in the 5.080-cm (2.00-in.)-diameter spherical acoustic resonance spectrometer described in Ref. 8 modified in accordance with the schematic block diagram shown in Fig. 1. The principal change is the inclusion in the flow loop of a gas headspace chamber (separator) consisting of a cylinder arranged horizontally to provide a relatively large contact interface between the major condensed phase (ca. 150 cm<sup>3</sup>) and the minor vapor phase (ca. 5 cm<sup>3</sup>). The low-dead volume strain-gauge diaphragm pressure monitor is connected by a capillary tube to the other components, which are all housed inside the stirred liquid temperature bath. The bath temperature and the data acquisition are under computer control as described in Ref. 8.

## 2.3. Procedure

To begin the experiments, the clean and evacuated apparatus heated to 323 K was charged with 150 cm<sup>3</sup> of stabilized oil from a piston pump, which was also heated to 323 K. This heating precaution was taken to avoid the possibility of phase separation in the oil at room temperature. The components of the apparatus were arranged so that the oil completely filled the resonator cavity and the double-acting piston-type circulating pump. There also was a layer of oil in the separator, as shown in Fig. 1.

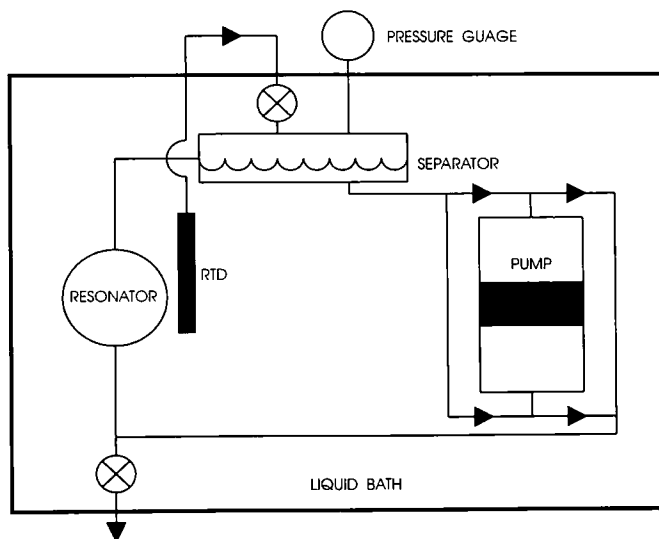


Fig. 1. Schematic diagram of the apparatus.

The light gas was then added, and blended, to reach the starting pressure, 0.2 MPa (2 bar). The procedure used for transferring the gas to the spectrometer without incurring phase change and composition variability involved maintaining the supply tank pressure well above the upper limit of the two-phase envelope, which occurs at about 12 MPa (120 bar), using external electric heaters and insulation wrap as needed. The single-phase fluid was delivered to the apparatus through a stainless-steel capillary tube which was continuously self-purged by the following stream. When the system pressure reached the target value, the gas supply was valved off and the circulating pump operated until equilibrium was achieved, as shown by a steady system pressure and more sensitively by a steady acoustic resonance signal. If more gas was needed to reach the desired equilibrium state, the above procedure was repeated. Following charging and equilibration, a resonance showing good resolution was chosen, and the data acquisition program was used to lock onto it. The temperature was slowly ramped (typically about  $0.2 \text{ mK} \cdot \text{s}^{-1}$ ) as the resonance frequency, temperature, and the system pressure were measured and stored to memory at frequent intervals.

The results were analyzed graphically and numerically to identify the first onset of phase separation corresponding to the initial precipitation of heavy components or the wax appearance point (WAP). Repeat runs were made to confirm the reproducibility of the measurements, and runs were made while increasing the temperature to investigate the re-resolution of the precipitated wax. Following collection of data at one gas charge, the system was heated to 323K and the additional gas was added and blended to reach the next higher charge. Data were then collected as before.

### 3. RESULTS

The initial experiments, all of which were made at low gas loadings, showed that the behavior of the sample was strongly dependent on its past history, especially if it were cooled substantially below the first wax appearance point. This was believed to be associated with a substantial phase separation accompanied by plating out of the wax on the apparatus walls, from which the kinetics of re-resolution were very slow. Once this behavior was recognized, the system was disassembled, cleaned and charged with a fresh sample. Afterward care was taken to avoid deep cooling of the system. If the temperature is not lowered more than a few degrees below the first evidence of wax formation, the crystals appear to remain suspended and return to solution on heating with essentially no hysteresis. Rapid heating or cooling, however, leads to irreproducible results in which the solution or precipitation lags behind the equilibrium

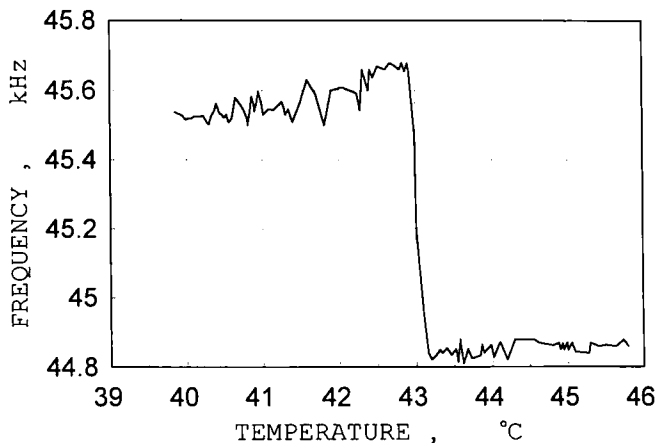


Fig. 2. Resonance frequency versus temperature for a cooling run with a 26-bar light gas loading.

value. We report here only the reproducible first wax appearance points of this oil when charged to five starting pressures in the range from 0.2 to 10 MPa (2 to 100 bar). The results are tabulated in Table II. Typical cooling curves for resonance frequency versus temperature and for system pressure versus temperature are shown for the 2.6 MPa (26 bar) loading run in Figs. 2 and 3. These figures illustrate the characteristically good sensitivity to change of the acoustic resonance method relative to conventional *PVT* measurements.

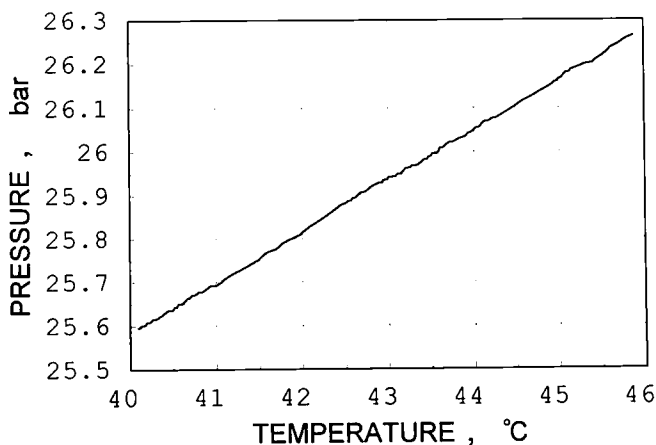
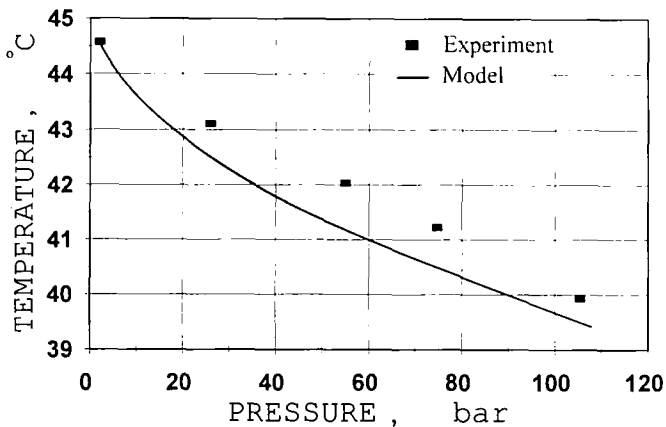


Fig. 3. System pressure versus temperature for a cooling run with a 26-bar light gas loading.

**Table II.** Pressure Dependence of Wax Appearance Point for Njord Well Fluid

Pressure (bar)	Temperature (°C)
1.96	44.58 ± 0.05
25.94	43.11 ± 0.05
55.01	42.04 ± 0.05
74.65	41.23 ± 0.1
105.42	39.94 ± 0.05

In Fig. 4 the wax appearance points determined acoustically are compared graphically with the predictions of the Norsk Hydro model [1]. Except for the case of the lowest gas loading (ca. 0.2 MPa) the measured WAP is higher than the model prediction by 1 K or less. Both methods show the effect of increased solubility of the heavy components (wax) caused by light gas loading of this North Sea crude oil. This work illustrates what is perhaps the major utility of the present experimental technique, namely, its use to acquire data on select systems to support the development of or test the reliability of predictive models.



**Fig. 4.** Comparison of acoustic determined wax appearance with the Norsk Hydro predictive model [1].

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