Influence of pressure on wax precipitation in crude oil with HP-µDSC

Introduction

The presence of paraffin hydrocarbons (C18 - C36) and naphthenic hydrocarbons (C30 - C60) in crude oil, diesels and biodiesels is known to cause wax precipitation at low temperature. Solidified waxes thickens the oil and allow to clog fuel filters and injectors in engines (in the case of diesels or biodiesels) and can cause flow assurance related problems as bad as full pipeline blockage (in the case of crude oil).
The prevention of precipitation requires a detailed characterization of the crude oil. On top of its simplicity and fast response, Differential Scanning Calorimetry (DSC) technique has the advantage of allowing operations at the high pressures that can be met in engines or pipelines.

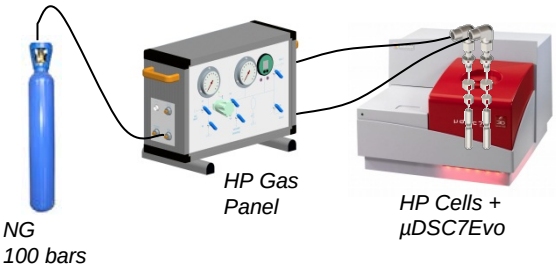

Figure 1 – Set-up for high pressure DSC tests

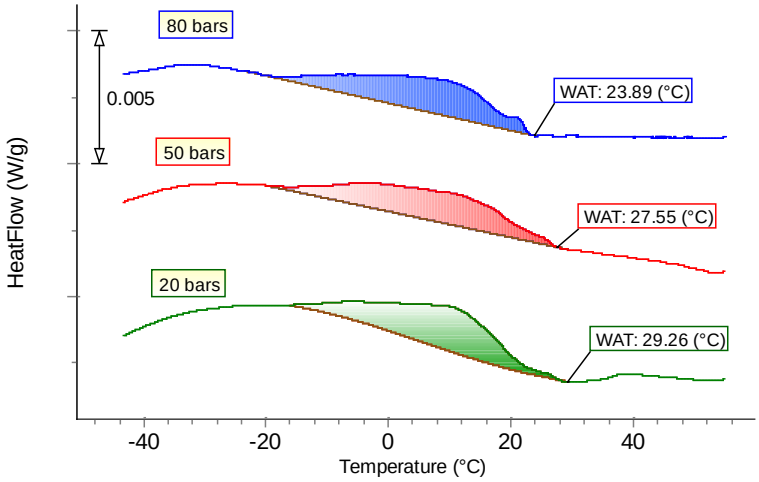

Figure 2 – Exothermic effects of wax precipitation

Experimental

187.9mg of crude oil were introduced in a µDSC7 Evo high pressure cell. The vessels were connected to the outputs of the high pressure gas panel. The input of the gas panel was connected to a 100 bars natural gas bottle. The set-up is schematized in fig 1.

Amount of Crude oil in sample cell (mg)	Pressure maintained in the cell (bar)
187.9	20, 50, 80

Table 1 – Used amounts of sample

Three experiments were run at 80 bars, 50 bars and 20 bars. After a conditioning operation at 60°C during 24hrs, the following temperature ramp was programmed :
- Cooling down from +60°C to -45°C at 0.5 K.min-1

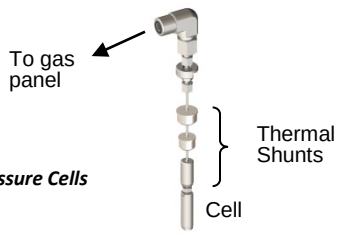

Figure 3 – µDSC7 evo High Pressure Cells

µDSC7 evo
-45 °C to 120°C

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Results

The highest temperature at which wax precipitation occurs in crude or heavy oils is called cloud point, wax appearance temperature (WAT) or wax precipitation temperature (WPT).

Pressure (bar)	Heat (J/g)	WAT (°C)
20	-4.442	29.26
50	-4.021	27.55
80	-4.057	23.89

Table 2 – results of peak integrations

This WAT is shown to be quite largely influenced by the pressure above the sample. It decreased by a value of 5.37°C between 20bars and 80bars.

Partial integral function of Calisto™ software allow to plot the percentage of converted wax vs. temperature.