



Design and test of a new high pressure phase equilibrium apparatus for highly corrosive mixtures of importance for natural gas



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ABSTRACT

A new static analytical apparatus for high-pressure phase equilibrium measurements has been designed and built. The new apparatus enables the measurement of vapor–liquid and liquid–liquid equilibria, which can operate at temperatures ranging from 225 K to 475 K and pressures up to 20 MPa. It is constructed in Titanium and alloy C276, being suitable for highly corrosive systems of interest for the gas industry (e.g., for hydrogen sulfide containing mixtures). The apparatus is equipped with two Rapid Online Sampling Injectors (ROLSI™) enabling the withdrawing of micro-samples without disturbing the equilibrium conditions. A gas chromatograph is connected to the apparatus for direct analysis of the phases' compositions. The quality and performance of the new apparatus has been evaluated by measuring a well reported system (carbon dioxide + methylcyclohexane).

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1. Introduction

Operations in oil and gas industry frequently require the use of high pressure conditions. Gas reservoirs are encountered at elevated pressures and temperatures. Gas sweetening, gas dehydration or hydrocarbon recovery are examples of high pressure processes in the gas industry.

As more sour gas fields are being developed, the need of accurate experimental data involving corrosive mixtures increases. Detailed thermodynamic data are essential for pipe transport and process design. In particular, the availability of experimental data of systems with hydrogen sulfide (H₂S) involved are very limited. The highly non-ideal behavior of these mixtures, particularly at moderate and high pressures, and the lack of predictive thermodynamic models (Hendriks et al., 2010), urges the development of reliable and safe experimental equipment for phase equilibrium

measurements. Knowledge of the liquid–gas distribution of a component is crucial for gas separation processes. In particular, the accurate description of the phase boundaries is essential to prevent the appearance of undesired phases, i.e., vaporization, (retrograde) condensation of streams during transportation, formation of hydrates, etc.

Experimental set-ups for high pressure phase equilibria can be classified as analytical systems, if the composition of the phases is determined, or as synthetic systems, if only the overall composition is known. Synthetic methods are based on the determination of phase transitions. The main advantages of these methods are their simpler design and operation, and the high accuracy of the measurements. Analytical methods, on the other hand, are more advantageous for multicomponent separation systems, because they allow the determination of tie-lines (Fornari et al., 1990; Dohrn et al., 2012). Isothermal analytical methods have been reported to produce reliable data when careful procedures are carried out (Christov and Dohrn, 2002).

In this work, the design and test of a new high pressure phase equilibrium apparatus is presented. The brand new analytical apparatus was designed to withstand highly corrosive mixtures at pressures up to 20 MPa and temperatures ranging from 225 K to 475 K. Preventing corrosion is a major challenge and a great

Abbreviations: F.S., full scale; TCD, thermal conductivity detector; FID, flame ionization detector; PID, proportional-integral-differential; CO₂, carbon dioxide; H₂S, hydrogen sulfide; MCH, methylcyclohexane; *T*, temperature; *P*, pressure; *x*_{CO₂}, molar composition of CO₂.

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concern for equipment integrity as well as operational safety. Therefore, the new apparatus has been constructed in titanium and alloy C276 (nickel-molybdenum-chromium and a small amount of tungsten), both exhibiting excellent corrosion resistance. These materials are commonly used in chemical and petrochemical industry.

The main challenge of analytical methods is the way the handling of the sampling is performed (Deiters and Schneider, 1986), which should not cause any disturbance of the equilibrium conditions. Most important is also that the samples should accurately represent the composition of the phases, without noticeably changing the overall compositions. In this new apparatus, Rapid Online Sampling Injectors (ROLSI™) are used for accurate sampling of phases. These injectors have been already successfully used for sampling systems at high pressures (Guilbot et al., 2000; Théveneau et al., 2006; Narasigadu et al., 2013; Nandi et al., 2013).

The capability of the new apparatus to measure accurate phase equilibrium data has been tested with a binary reference system that was extensively determined at various pressures, temperatures and compositions using the Cailletet apparatus (De Loos et al., 1986). The Cailletet apparatus is recognized as one of the most accurate experimental methods for measuring high pressure phase equilibria, but unfortunately unsuitable for measurement of systems containing H₂S. Therefore, we chose carbon dioxide (CO₂) + methylcyclohexane (MCH) as binary reference system (Nasrifar et al., 2003). Knowledge on the solubility of CO₂ + MCH system is crucial for understanding the inhibition of hydrate formation when insoluble organic solvents such as MCH are added to the water (H₂O) + CO₂ systems (Mooijer-van den Heuvel et al., 2001).

2. Apparatus and experimental procedure

2.1. Overview

The general view of the high-pressure phase equilibrium apparatus is presented in Fig. 1. This system comprises six main parts: (1), gas pressure vessels; (2), variable volume pump; (3), high-pressure equilibrium cell; (4), ROLSITM IV sampling system; (5), gas chromatograph; and (6), vacuum system. The system is equipped with 19 valves made of alloy C276, provided by Top Industrie. The valves are highly resistance to corrosion and their operating upper limit pressure is 100 MPa.

2.1.1. Gas pressure vessels

The gas dosing system is composed of three titanium pressure

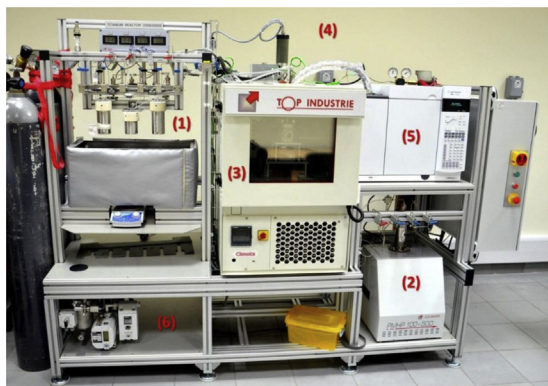


Fig. 1. General view of the Titanium high-pressure phase equilibrium apparatus. 1), gas pressure vessels; (2), variable volume pump; (3), high-pressure equilibrium cell; (4), ROLSITM IV sampling system; (5), gas chromatograph; and (6), vacuum system.

vessels (Fig. 2). They are designed to operate at pressures up to 20 MPa, with a pressure limit of 30 MPa. Two of the vessels, with an internal volume of 191 cm³, are used to load two different gases. These vessels are connected to a third vessel with an internal volume of 111 cm³, connected to the equilibrium cell. In this vessel, the two gases can be mixed. The gas dosing system can be submerged in a temperature controlled bath (e.g. water, ethanol or oil bath, depending on the working temperature range) to allow thermal stabilization and to prevent condensation when the gas is expanded to another vessel. The pressure of the gas pressure vessels is measured by three pressure transmitters (Keller, PA35X HTT-200 bar), with an accuracy of 0.2% full scale (F.S.).

2.1.2. Variable volume pump

A high precision variable volume pump (Top Industrie, model PMHP100 – 500) is used to load the liquid solution into the equilibrium cell, and can operate up to 50 MPa. The exact quantity of liquid introduced into the cell is determined by the displacement of the piston with an accuracy of 0.05%.

2.1.3. The high-pressure equilibrium cell

The equilibrium cell, constructed in titanium and with an internal volume of 50 cm³, can operate up to 20 MPa and in the temperature range between 225 K and 475 K. The titanium high-pressure equilibrium cell is presented in Fig. 3.

The cell consists of two separate segments screwed together. This construction enables the maintenance and cleaning of the interior of the cell. Two sapphire windows on the side of the cell allow the visual observation of the phases inside. The equilibrium cell is connected with the mixing vessel, with a gas inlet/drain connection and with the variable volume pump and the vacuum pump. A platinum stirrer, operated by an external magnetic rotor, is used to agitate the sample inside the cell, enhancing the mixing of the phases and reducing the time to attain equilibrium pressure. The capillary tubes from two ROLSITM IV samplers are inserted from



Fig. 2. Two 191 cm³ and one 111 cm³ pressure vessels of the gas dosing system and their connections above a temperature controlled bath.

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