

Phase Equilibria Relevant to Acid Gas Injection: Part 2—Aqueous Phase Behaviour

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Abstract

The purpose of this paper is to review the literature for the available experimental data and briefly survey methods for calculations of the aqueous equilibria of acid gas mixtures; notably, the water content of the gas and liquified acid gas. In addition to water, this study will include the following components: hydrogen sulfide, carbon dioxide, methane, ethane, and propane.

The design engineer should be fully informed of the capabilities of the model selected to perform the calculations. If uncertain, it is wise to verify the chosen model by comparing it with experimental data. However, as will be demonstrated, the design engineer should be critical when interpreting the available data.

Acid Gas Injection

As was stated in the first part of this paper, acid gas injection has become an important method for dealing with unwanted acid gas.

A common approach in the design of an acid gas injection scheme is to take advantage of the thermodynamics of these systems. The water content of acid gas mixture has a minimum as a function of the pressure. The design of an acid gas injection project should attempt to take advantage of this minimum, in order to eliminate the need for dehydration⁽¹⁾.

Water Content

An essential aspect of the design of an acid gas injection scheme is the water content of the acid gas mixture. In addition, it is important to know the effect of the state (gas or liquid) of the acid gas on the water content. Table 1 lists experimental investigations into the water content of mixtures containing hydrogen sulfide and/or carbon dioxide.

The study of Selleck et al.⁽²⁾ is considered the benchmark investigation of the system hydrogen sulfide + water. They published tables of smoothed data, which are commonly quoted in the literature. However, these tables are based on relatively few and scattered experimental data points. Carroll and Mather⁽³⁾ re-evaluated the phase behaviour in this system, presenting a clearer picture of the equilibria and accurately reflecting all of the available experimental data.

There have been many investigations of the water content of CO₂-rich fluids. In general, there is reasonable agreement amongst the various sets of data in the low and moderate pressure regions. The benchmark investigation of the phase behaviour in the system carbon dioxide + water was that of Wiebe and Gaddy⁽⁴⁻⁶⁾.

Finally, the author of this paper has performed thorough

reviews of the literature, and is unaware of any experimental data for the water content for binary mixtures of H₂S + CO₂ in the public domain. Such data, if available, would be very useful.

There have been several experimental investigations into the water content of hydrocarbons. Table 2 lists those of interest in this study. In this paper, we are not strictly interested in the water content of hydrocarbons, but in acid gas mixtures containing hydrocarbons. We require a model that accurately predicts the water content of hydrocarbons in order to have the confidence that it will work for multicomponent mixtures. Those are the reasons why they are included here.

Solubility

The topic of the solubility of these gases in water is also an immense subject. Although it too is of some importance to the design of an acid gas injection scheme, it will not be discussed here. Recent reviews of the solubility of carbon dioxide in water^(7, 8) and hydrogen sulfide in water^(9, 10) are available to those interested in the subject.

Calculation Methods

Equations of state, which are widely used for petroleum systems, cannot be used for aqueous systems. As a first criterion for their application, an equation of state must accurately predict the vapour pressure of the pure components. Neither the original form of the SRK⁽¹¹⁾ nor the PR⁽¹²⁾ accurately reproduces the vapour pressure of water, so one must resort to a modified form, such as the one proposed by Stryjek and Vera⁽¹³⁾, the PRSV equation. However, there are other similar modifications available for both the PR and SRK equations.

The next problem with calculating the phase behaviour in aqueous systems is that simple mixing rules are inadequate. This, too, can be overcome, at least in theory. Without being over-simplistic, this requires new mixing rules. Basically, the mixing rule must be phase specific. In order to be phase specific, the mixing rule must include a density effect, a composition effect, or both. Simply having a mixing rule that is temperature-dependent will not work, because the temperature of the phases is all the same. The same is true of the pressure. Wong and Sandler⁽¹⁴⁾ proposed a mixing rule that has become quite popular, but there are many others available.

Having said that, a properly constructed equation of state can be used for calculating the multiphase equilibrium in systems containing acid gas + water systems. However, the design engineer would be wise to confirm that the modelling software they are

TABLE 1: Experimental investigations of the water content of mixtures containing hydrogen sulfide and carbon dioxide.

Gas	Temperature (° C)	Pressure (MPa)	Reference
H ₂ S	5 to 60	up to 0.50	Wright and Maass ⁽²³⁾
	37 to 171	2.7 to 35	Selleck et al. ⁽²⁾
	90 to 150	1.5 to 3.5	Lee and Mather ⁽²⁴⁾
	37 to 315	up to 10	Gillespie et al. ⁽²⁵⁾
CO ₂	25 to 75	0.1 to 71.0	Wiebe and Gaddy ⁽⁶⁾
	25 to 100	1.7 to 5.1	Coan and King ⁽²⁶⁾
	100 to 200	0.2 to 5.0	Zawisza and Malesinska ⁽²⁷⁾
	16 to 260	0.7 to 13.8	Gillespie et al. ⁽²⁵⁾
	-28 to 25	0.7 to 13.8	Song and Kobayashi ⁽²⁸⁾
	100 to 200	0.3 to 8.1	Müller et al. ⁽²⁹⁾
H ₂ S + CH ₄	70	1.3 to 10.3	Lukacs and Robinson ⁽³¹⁾
	54 and 71	6.9 to 10.3	Maddox et al. ⁽³²⁾
CO ₂ + CH ₄	37 and 71	6.9 and 13.8	Maddox et al. ⁽³²⁾
	15 to 50	5.7 to 13.8	Song and Kobayashi ⁽³³⁾
H ₂ S + CO ₂ + CH ₄	37 to 177	4.8 to 18.2	Huang et al. ⁽³⁴⁾
	37	7.6 and 13.1	Maddox et al. ⁽³²⁾
H ₂ S + CO ₂ + CH ₄ + C ₃ H ₈	49 and 93	1.4 to 69	Ng et al. ⁽³⁵⁾

TABLE 2: Experimental investigations of the water content of mixtures containing light hydrocarbons.

Gas	Temperature (° C)	Pressure (MPa)	Reference
CH ₄	37 to 240	up to 70	Olds et al. ⁽³⁶⁾
	70	1.2 to 10.3	Lukacs and Robinson ⁽³¹⁾
	25 to 100	2.2 to 10	Rigby and Prausnitz ⁽³⁷⁾
C ₂ H ₆	37 to 240	up to 70	Reamer et al. ⁽³⁸⁾
	25 to 100	2.2 to 5.1	Coan and King ⁽²⁶⁾
	-30 to 30	?	Parrish et al. ⁽³⁹⁾
	-33 to 32	2.5 to 4.8	Song and Kobayashi ⁽⁴⁰⁾
C ₃ H ₈	37 to 150	up to 20	Kobayashi and Katz ⁽⁴¹⁾
	312 to 387	up to 200	de Loos et al. ⁽⁴²⁾
	-30 to 30	?	Parrish et al. ⁽³⁷⁾
	-37 to 27	0.6 to 1.1	Song and Kobayashi ⁽⁴⁰⁾
CH ₄ + C ₂ H ₆	32 to 60	4 to 20	Villarreal et al. ⁽⁴³⁾
C ₂ H ₆ + C ₃ H ₈	-30 to 30	?	Parrish et al. ⁽³⁷⁾
	-17 to 24	4.1	Song and Kobayashi ⁽⁴⁰⁾

- the pressure of these measurements is not stated, but they indicate that they are at the “vapor pressure” of the hydrocarbon.

using is sufficiently accurate for their design. To verify the model, the design engineer should compare the predictions with the experimental data discussed earlier.

An alternative to using equations of state is to use a two-fluid approach. For example, a Henry’s law approach can be used for the aqueous phase, and an equation of state for the non-aqueous phases. This method has been successfully implemented in *AQUAlibrium*, a commercially available software package. Results presented in this paper for equilibria involving water were calculated using *AQUAlibrium*.

The first series of calculations is intended to show the accuracy of *AQUAlibrium*. Similar accuracy can be anticipated from any well-constructed, thermodynamically consistent model. But, as was mentioned earlier, the design engineer is well advised to confirm that the model chosen is indeed applicable for this purpose.

In general, the simple methods used for estimating the water content of sweet gas should not be used for acid gas. For example,

the McKetta chart provided in the GPSA Engineering Data Book⁽¹⁵⁾ is not accurate for acid gases, especially under pressure. As will be demonstrated, the water content of acid gases differs from that of sweet gases. There are corrections provided in the GPSA Engineering Data Book for sour gas, but even they are insufficient for acid gases.

Hydrogen Sulfide

Figure 1 shows three isotherms for mixtures of water and hydrogen sulfide. This figure is difficult to interpret. The calculations show three branches. The lower branch is the water content of the gas. This curve indicates that the water content of the gas is a strong function of pressure—as the pressure increases, the water content decreases. The horizontal broken line is a three-phase point, the three phases being an aqueous liquid, an H₂S-rich liquid, and a vapour. The third branch, which is very steep, is the

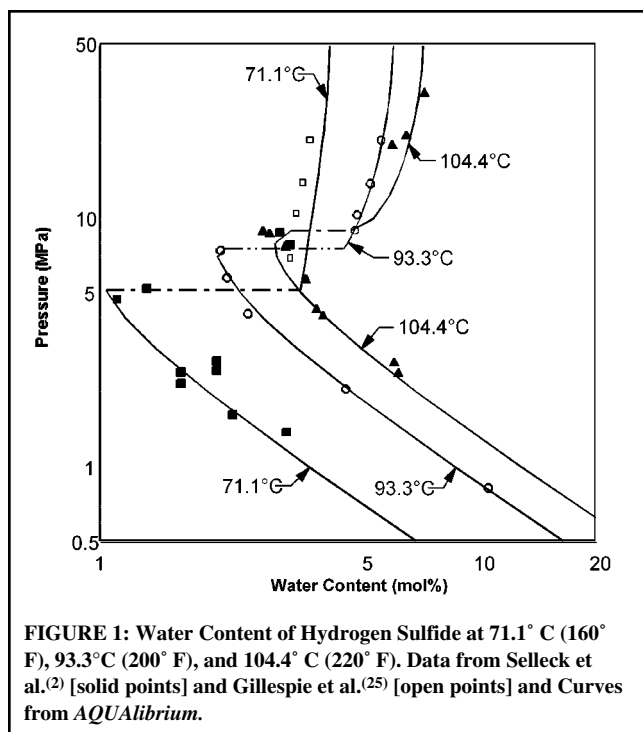


FIGURE 1: Water Content of Hydrogen Sulfide at 71.1° C (160° F), 93.3° C (200° F), and 104.4° C (220° F). Data from Selleck et al.⁽²⁾ [solid points] and Gillespie et al.⁽²⁵⁾ [open points] and Curves from *AQUAlibrium*.

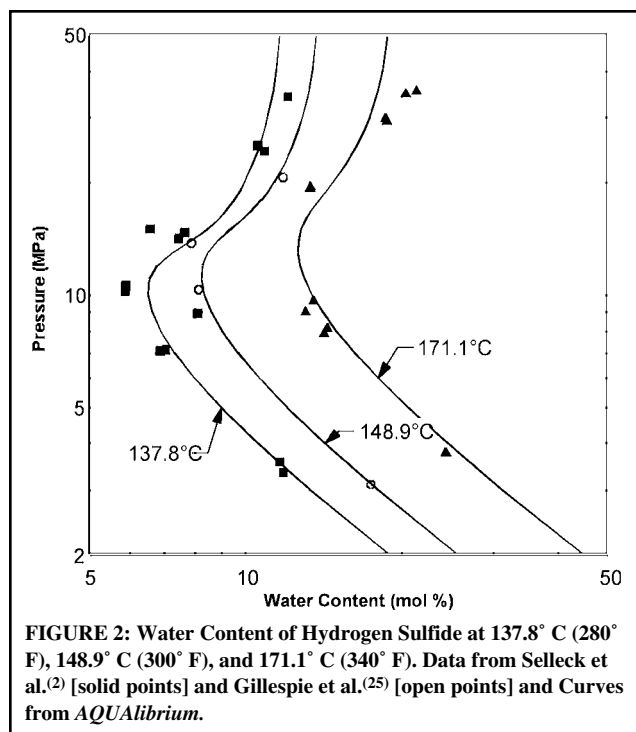


FIGURE 2: Water Content of Hydrogen Sulfide at 137.8° C (280° F), 148.9° C (300° F), and 171.1° C (340° F). Data from Selleck et al.⁽²⁾ [solid points] and Gillespie et al.⁽²⁵⁾ [open points] and Curves from *AQUAlibrium*.

water content of liquid H₂S. A small region representing the non-aqueous vapour-liquid equilibrium for a mixture of H₂S + H₂O, and lean in water, is not shown. This region would extend from the three-phase point to the vapour pressure of pure H₂S at the zero water-content axis.

Note that all of these isotherms have a three-phase point, represented by the horizontal, broken line. This includes a three-phase point at 104.4° C, which Selleck et al.⁽²⁾ believed did not exist, probably because this temperature is greater than the critical point of pure H₂S (100° C). However, its existence was demonstrated by Carroll and Mather⁽³⁾. Note the significant scatter in the experimental data, and in particular, the data of Selleck et al. This presents a different picture of the phase equilibrium than their often-quoted smoothed data.

This plot demonstrates the aforementioned problem with the data of Selleck et al.⁽²⁾ At this temperature, they measured only three points for the water content of H₂S, only one of which is in the vapour region, whereas their smoothing has eleven points.

Figure 2 depicts three additional isotherms for the system H₂S + H₂O. All of these are for temperatures where an H₂S-rich liquid does not exist. The prominent feature of these curves is the minimum in the water content. For these isotherms, the minimum occurs between 10 and 15 MPa, but it is a function of the temperature. Again, note the significant scatter in the experimental data, particularly those of Selleck et al.⁽²⁾

Admittedly, *AQUAlibrium* is not a perfect fit of the experimental data for the binary system H₂S + H₂O. However, it is a good compromise, considering the scatter in the raw data available in the literature.

Carbon Dioxide

The phase behaviour in the system CO₂ + H₂O is qualitatively the same as H₂S + H₂O. Therefore, a detailed discussion of the nature of the equilibria for this system will not be presented. One significant difference between the phase equilibria for the two systems is that a CO₂-rich liquid does not exist for temperatures greater than about 31° C, whereas for H₂S, the non-aqueous liquid forms up to about 106° C.

Figure 3 shows the water content of carbon dioxide for five temperatures. Experimental data from five sources are also shown on this plot. Similar to the supercritical H₂S, the prominent feature on these plots is the minimum in the water content.

Figure 4 shows the water content of liquified and supercritical

CO₂ for five isotherms (the critical point of CO₂ is 31° C and 7.38 MPa). For the three lowest temperatures, the curves extend from the three-phase pressure. The other two curves are at temperatures where a CO₂-rich liquid does not form. These two isotherms do, however, exhibit the minimal behaviour shown in the previous figure. This region has been omitted for clarity.

For the isotherms shown in the two figures, *AQUAlibrium* is a good fit of the experimental data, with the exception of a few points. This includes the critical region, which is notorious as a region where phase equilibrium calculations are difficult.

Hydrocarbons

Although we are not specifically interested in the water content of hydrocarbons in this study, they are presented for two reasons. The first of these is, as was mentioned earlier, to demonstrate the accuracy of the software. The second reason is to show how the behaviour of hydrocarbons differs from that of the acid gas components.

Figure 5 shows three isotherms for the water content of methane. Note that for methane, the water content is a continually decreasing function of the pressure. This is what one would expect, but it differs from the behaviour shown earlier for acid gas mixtures.

Hydrates

Hydrates are ice-like solids that form in the presence of water and a relatively small molecule. The water molecules form a hydrogen-bonded lattice that is stabilized by the presence of the "guest" molecule. Hydrates form at temperatures greater than the freezing point of water and, therefore, a solid phase can form where one would not expect one to exist. Hydrates are notorious in the natural gas business for plugging flow lines and process equipment.

Of the components commonly found in natural gas, none forms a hydrate more easily than hydrogen sulfide. The hydrate of H₂S forms at the lowest pressure, and persists to the highest temperature. Carbon dioxide is also a hydrate former. Thus acid gas mixtures are notorious for forming hydrates.

The literature for the formation of hydrates in the system H₂S has been thoroughly reviewed by Carroll and Mather⁽¹⁶⁾. This review covers all investigations back to the middle of the 19th

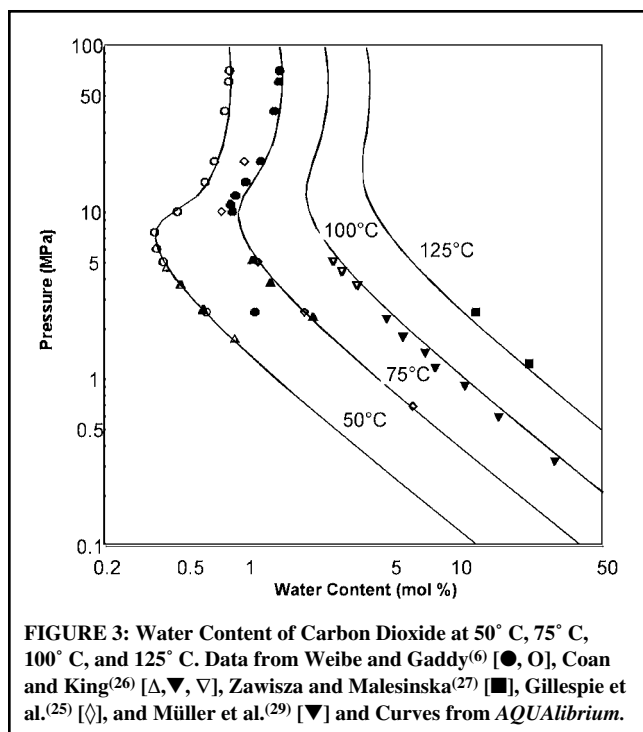


FIGURE 3: Water Content of Carbon Dioxide at 50° C, 75° C, 100° C, and 125° C. Data from Weibe and Gaddy⁽⁶⁾ [●, ○], Coan and King⁽²⁶⁾ [△, ▽], Zawisza and Malesinska⁽²⁷⁾ [■], Gillespie et al.⁽²⁵⁾ [◇], and Müller et al.⁽²⁹⁾ [▼] and Curves from AQUALibrium.

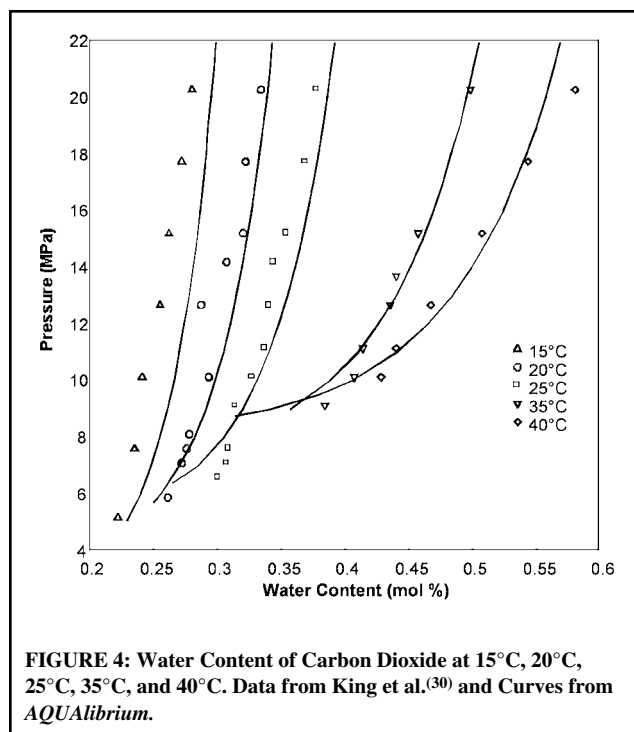


FIGURE 4: Water Content of Carbon Dioxide at 15° C, 20° C, 25° C, 35° C, and 40° C. Data from King et al.⁽³⁰⁾ and Curves from AQUALibrium.

century. A good review of the carbon dioxide hydrate was presented by Bakker et al.⁽¹⁷⁾ For studies of hydrates in other systems, the reader is referred to the tome of Sloan⁽¹⁸⁾.

Inhibition

In the natural gas industry, there are three common approaches to combating hydrates: (1) dehydration, (2) the use of heat, and (3) inhibition with chemicals, usually methanol. The philosophy behind dehydration is that, if there is insufficient water present, then a hydrate cannot form. Essentially, heat is used to keep the fluid at a temperature above that at which a hydrate can form. Chemicals, such as alcohols or glycols, can be used to depress the temperature at which a hydrate forms.

Ng and Robinson⁽¹⁹⁾ presented some data for the hydrate-forming conditions of $H_2S + CO_2 + CH_4$ in the presence of methanol.

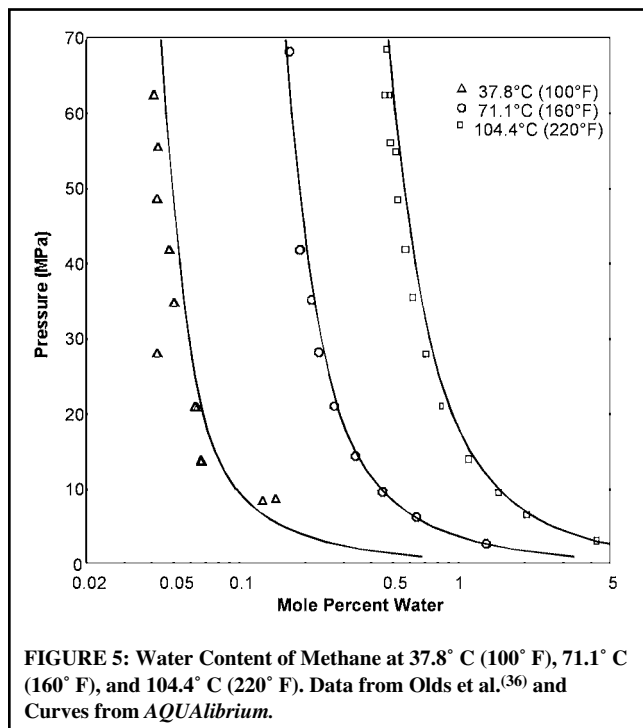


FIGURE 5: Water Content of Methane at 37.8° C (100° F), 71.1° C (160° F), and 104.4° C (220° F). Data from Olds et al.⁽³⁶⁾ and Curves from AQUALibrium.

In addition to the inhibiting effect of methanol, they observed another interesting phenomenon. Since H_2S and CO_2 have significant solubilities, the methanol affected the composition of the gas. The gas was leaner in these components than the feed gas. Since the acid gas components are significant contributors to the hydrate formation, their removal from the non-aqueous phases can magnify the inhibiting effect.

Calculation Methods

A commonly employed method for doing rapid hydrate calculations is the Katz K-factor charts⁽¹⁵⁾. When applied to the pure acid gas components, these charts are surprisingly accurate from the lower quadruple point to the upper quadruple point. However, they should not be used outside this range. Their accuracy is reduced considerably when applied to mixtures of acid gases. In general, their use for such mixtures is not recommended.

Computer methods are based on the theory of van der Waals and Platteeuw⁽²⁰⁾. Essentially, these use a statistical thermodynamic model to estimate the conditions at which a hydrate will form. Most modern calculation packages are based on the theory of van der Waals and Platteeuw, although modifications have been included to account for the pressures and phases of interest to natural gas industry. Two notable examples are the models of Parrish and Prausnitz⁽²¹⁾ and Ng and Robinson⁽²²⁾. These models, and the software packages developed from them, can successfully model the hydrate forming conditions in acid gas mixtures.

Concluding Remarks

In the design of an acid gas injection scheme, the design engineer must consider a wide range of phase equilibria. The presence of water makes the phase equilibria more difficult. Engineers must be prepared to handle these calculations with the proper set of calculation tools that have been constructed using the best available experimental data.

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