NMR Study on the Reactivity of Hydrogen Sulfide in Aqueous Amine Solutions

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Abstract

Removing hydrogen sulfide (H_2S) from gas streams is a critical task in many industrial processes, e.g. in natural gas cleaning where it is generally carried out together with the removal of carbon dioxide (CO_2) by reactive absorption. The most common solvents for the reactive CO_2 absorption are aqueous amine solutions, sometimes with alcohols as co-solvents. The sour gases H_2S and CO_2 dissociate in these basic solutions, leading to high solubilities, which are further enhanced by chemical reactions of CO_2 with many amines. It is usually assumed that H_2S only dissociates in these solvents, but does not react chemically as CO_2 does; however, no experimental proof of this hypothesis has been given in the literature so far. Therefore, we have carried out an NMR study of the speciation of aqueous amine solutions containing N-methyldiethanolamine (MDEA), monoethanolamine (MEA), piperazine (PZ), and ethanol (EOH), which were loaded with H_2S at pressures between 2.7 bar and 4.8 bar at 323.15 K. To identify possible chemical reactions of H_2S with the solvents, the resulting NMR spectra were compared with the NMR spectra of the unloaded aqueous amine solutions. The dissociation of H_2S in the basic amine solutions leads to a protonation of the amines and strong NMR peak shifts, but no reaction products of H_2S with MDEA, MEA, PZ, and EOH were found. We expect, that this is also true for other amines and alcohols and confirms the hypothesis that the solubility of H_2S in the studied solvent class is not affected by chemical reactions with the solvents.

Introduction

Reactive gas absorption is often used to purify gas streams containing acid gases such as carbon dioxide (CO_2) or hydrogen sulfide (H_2S).[1–4] Large scale applications include the cleaning of natural gas, refinery gas, biogas, and synthesis gas. Mostly, aqueous amine solutions are used as solvents in these processes.[1,5–7] Sometimes also co-solvents are added, in particular alcohols.[8,9] In these solutions, the presence of the basic amine leads to a dissociation of H_2S

and CO_2, which strongly increases the solubility. Furthermore, CO_2 reacts with primary and secondary amines to form carbamates[10–13] and with alkanolamines and alcohols to alkylcarbonates.[14–16] Both the dissociation and the chemical reactions have to be considered in the modelling of reactive absorption.[17–20] In these models, it is usually assumed that H_2S dissociates, but does not chemically react with the solvents.[21–24] However, to the best of our knowledge, this hypothesis has never been tested experimentally. Maddox et al.[25] report on a study of non-aqueous mixtures of H_2S and amines by nuclear magnetic resonance (NMR) spectroscopy and have found no evidence for reaction products, even though they speculate about a Lewis acid - base complex. As H_2S is known to react with various substrates[26–29] we have decided to clarify experimentally whether or not chemical reactions between H_2S and aqueous amines and alcohols occur under the conditions relevant to industrial absorption processes. NMR spectroscopy is the method of choice for such a study and has been widely used for elucidation of species distributions in the reactive mixtures involved in the absorption of sour gases in aqueous amines.[12,14,30–32]

Experimental Section

Chemicals and Sample Preparation. Table 1 gives an overview of the chemicals that were used in the present work and their purity as specified by the supplier. All chemicals were used as received. The supplier of H_2S reported the following details on the impurities: carbonyl sulfide (COS) < 3000 ppm, methane (CH_4) < 500 ppm. Ultrapure water (W) was taken from a Starpure's OmniaPure UV/UF-TOC water treatment system with a resistivity of 18.2 MΩ cm.

Table 1: Overview of the chemicals that were used and their purity.

Component	Abbreviation	Supplier	Purity
Hydrogen sulfide	H_2S	Air Liquide	$\geq 99.5\%$
Monoethanolamine	MEA	Sigma-Aldrich	$\geq 99.0\%$
N-methyldiethanolamine	MDEA	Sigma-Aldrich	$\geq 99.0\%$
Piperazine	PZ	Sigma-Aldrich	$\geq 99.0\%$
Ethanol	EOH	Merck	$\geq 99.9\%$

Aqueous stock solutions of the solvents were prepared gravimetrically using a laboratory balance (Mettler-Toledo XSR225DU) with an accuracy of $\pm\,10^{-5}\,\mathrm{g\,g^{-1}}$. From these solutions, samples were taken with a syringe and filled into high pressure NMR sample tubes (S-5-500-HW-EX3-HPV-7, Norell). The composition of the stock solutions is given in Table 2.

Table 2: Composition of the studied (gas-free) solvents.

System	Component i	x_i / $\mathrm{g\,g^{-1}}$
MEA + W	MEA	0.3822
MDEA + PZ + W	MDEA	0.3786
	PZ	0.0996
MDEA + EOH + W	MDEA	0.3829
	EOH	0.3837

Loading of the solvents with H_2S. In this work, a custom-built apparatus for loading aqueous amine solutions with H_2S in high pressure NMR sample tubes was used, see Figure 1. It is similar to equipment we have used in previous work,[14] but was adapted to the needs of experiments with the highly corrosive and toxic gas H_2S. The gas loading procedure is only briefly described here, for more detailed information see Ref.[14] The sample tubes contain the unloaded solvent and are connected to a cylinder (B1) that is filled with H_2S from a storage cylinder (B2). An emergency shut off valve (V4) is installed near the outlet of the H_2S reservoir (B1). The samples were pressurized and thermostated for at least 168 h. This time is well above the time needed for the establishment of the gas-liquid equilibrium and also well above residence times in typical absorption equipment. It was chosen in order to check, whether over long times at elevated temperatures side reactions would occur. The composition of the loaded liquid (as well as that of the gas phase above it) is fully specified by the composition of the unloaded solvent, the pressure, and the temperature. For literature on the solubility of H_2S in the studied solvents, see Refs.[21,33–37] After equilibration, the sample tubes were removed from the thermostat and inserted into the NMR spectrometer where they were re-equilibrated to the desired temperature before the measurement was started.

After the experiment, the apparatus was flushed with nitrogen, and the waste was fed into the vessel (B3) containing a 6 M aqueous sodium hydroxide solution to bind all H_2S. The entire set-up was installed in a fume hood which was equipped with an H_2S sensor.

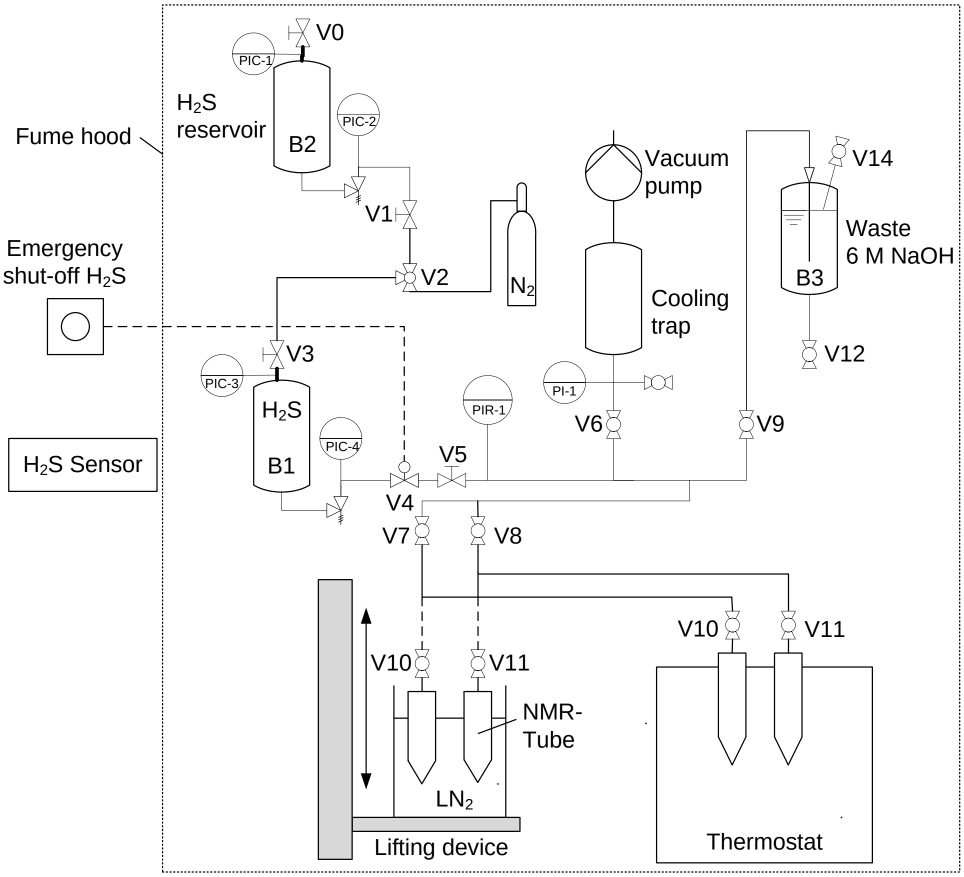

Figure 1: Set-up for loading the solvent in the NMR sample tubes with H_2S at the desired temperature and pressure.

NMR measurements. The NMR measurements were carried out in a similar way as in our previous work[14] and are therefore only briefly described here. An NMR spectrometer with a magnetic field strength of 9.4 T was used, which was equipped with a double resonance broadband probe with cryogenically cooled electronics (Bruker Biospin: Ascend 400, Avance III HD 400, CryoProbe Prodigy). The temperature of the spectrometer was controlled by a thermostated nitrogen gas flow, the temperature was calibrated using a platinum resistance thermometer (Pt-100), which was calibrated in our laboratory using a certified standard. The uncertainty of the temperature measurement is 0.5 K. Quantitative ^{1}H and ^{13}C NMR measurements were carried out at 323.15 K with the following settings: ^{1}H NMR measurements were carried out with ^{13}C decoupling using an excitation pulse angle of 10 degree to avoid radiation damping, 45 s relaxation delay, and 128 scans. ^{13}C NMR measurements were carried out using ^{13}C inverse gating and ^{1}H decoupling with 120 s relaxation delay and 256 scans. Baseline and phase correction of the spectra were performed manually with MestReNova (Mestrelab Research).

Results and Discussion

Chemistry of H_2S-loaded aqueous amine solutions

For understanding and modeling the H_2S absorption process, information on the chemical processes in H_2S-loaded aqueous amine solutions is essential. In these solutions H_2S dissociates, which also influences the dissociation of water and the protonation of the amines. This is summarized for aqueous solutions of the amines MEA, MDEA, and PZ in (I) - (VII).[4,12,35–41]

$$H_2O \rightleftharpoons H^+ + OH^- \tag{I}$$

$$H_2S \rightleftharpoons HS^- + H^+ \tag{II}$$

$$HS^- \rightleftharpoons S^{2-} + H^+ \tag{III}$$

$$MEA + H^+ \rightleftharpoons MEAH^+ \tag{IV}$$

$$MDEA + H^+ \rightleftharpoons MDEAH^+ \tag{V}$$

$$PZ + H^+ \rightleftharpoons PZH^+ \tag{VI}$$

$$PZH^+ + H^+ \rightleftharpoons PZ(H^+)_2 \tag{VII}$$

To the best of our knowlegde, no chemical reactions of H_2S with these or other amines used for chemical absorption have been reported in the literature. All reactions mentioned above are dissociations and extremely fast. The pKa values of these dissociations in water are known.[39,42] The dissociation of H_2S in the basic amine solutions leads to a protonation of the amines. At the studied conditions, the predominant sulfuric species is HS^-.

System H_2S + MEA + W

^1H NMR spectra and ^{13}C NMR spectra of a H_2S loaded aqueous MEA solution and reference spectra of the unloaded solvent MEA + W are shown in Figures 2 and 3, including the NMR peak assignment. Only peaks resulting from MEA and W were observed. There is no evidence for reaction products of H_2S and MEA. The observed peak shifts upon loading the solution with H_2S are due to the change in the pH value. Due to the very fast exchange any peak arising from protons bound to sulfur is not discernible from the protons in water. Hence, the signals from HS^- and H_2O are merged into a single large peak. Molecular H_2S is practically not present at the studied conditions.

The experiments were repeated with a higher H_2S loading at 4.8 bar, but gave the same results.

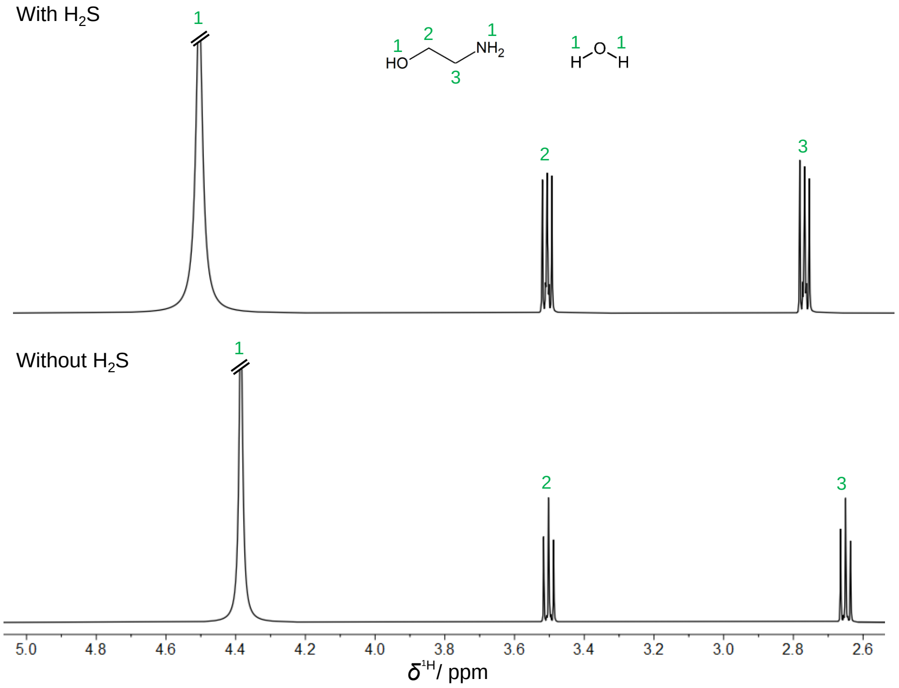

Figure 2: ^{1}H NMR spectra of a H_2S loaded aqueous MEA solution (top) and of the corresponding unloaded aqueous MEA solution (bottom). Solvent composition, see Table 2, solvent saturated with H_2S at 323.15 K and 3.4 bar.

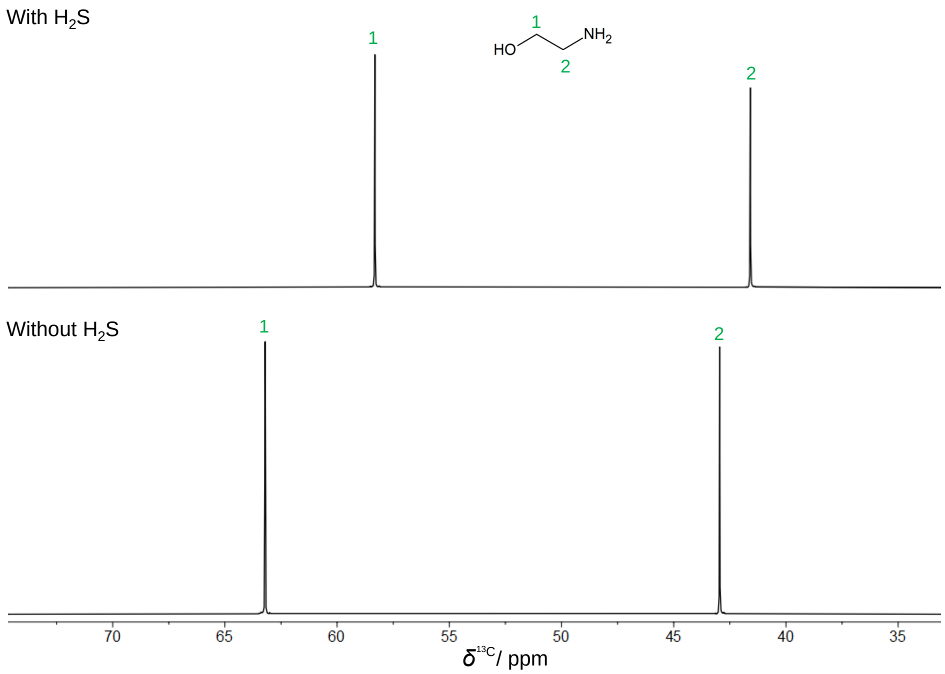

Figure 3: ^{13}C NMR spectra of a H_2S loaded aqueous MEA solution (top) and of the corresponding unloaded aqueous MEA solution (bottom). Solvent composition, see Table 2, solvent saturated with H_2S at 323.15 K and 3.4 bar.

System H_2S + MDEA + PZ + W

^{1}H and ^{13}C NMR spectra of a H_2S loaded aqeuous solution containing the amines MDEA and PZ and corresponding spectra of the unloaded soution are shown in Figures 4 and 5 together with the peak assignment. Again peak shifts upon H_2S loading, but no new peaks are observed. There is again no evidence for reaction products of H_2S with MDEA or PZ. The experiments were repeated with a higher H_2S loading at 4.7 bar, but gave the same results.

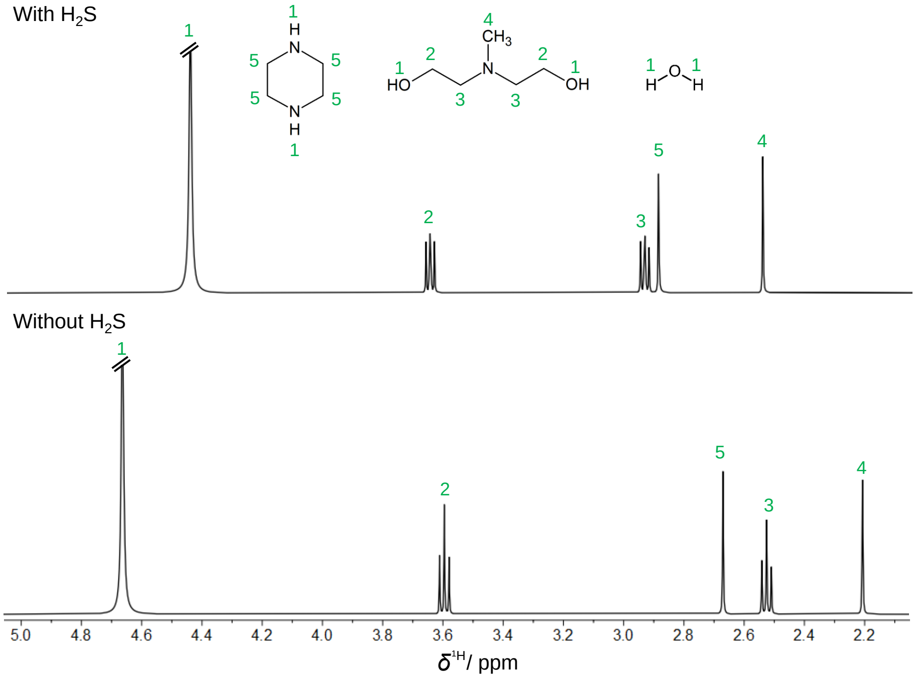

Figure 4: ^{1}H NMR spectra of a H_2S loaded aqueous MDEA + PZ solution (top) and of the corresponding unloaded aqueous MDEA + PZ solution (bottom). Solvent composition, see Table 2, solvent saturated with H_2S at 323.15 K and 3.2 bar.

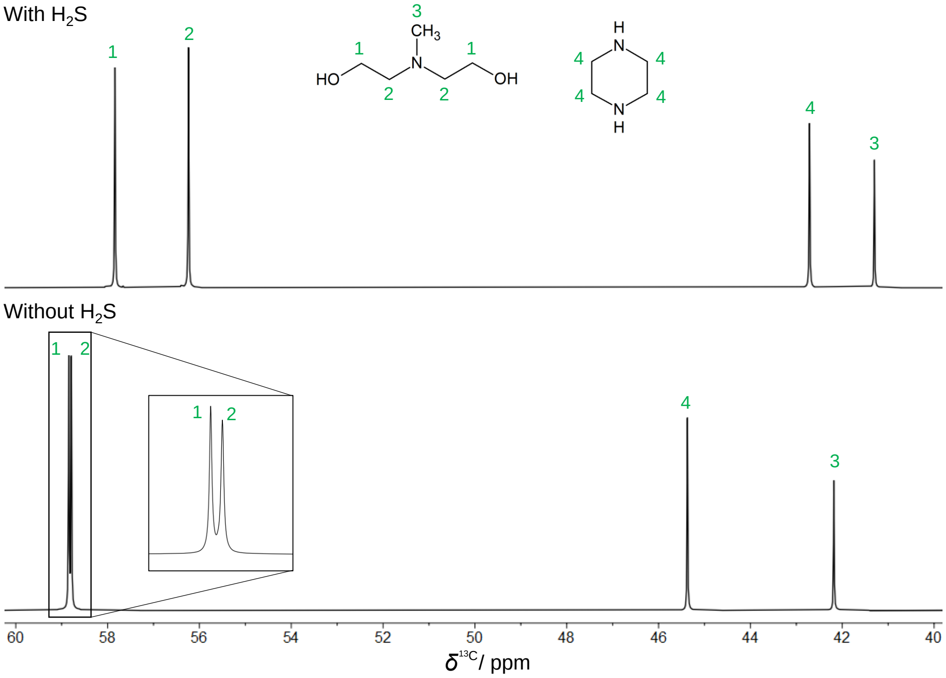

Figure 5: ^{13}C NMR spectra of a H_2S loaded aqueous MDEA + PZ solution (top) and of the corresponding unloaded aqueous MDEA + PZ solution (bottom). Solvent composition, see Table 2, solvent saturated with H_2S at 323.15 K and 3.2 bar.

System H_2S + MDEA + EOH + W

^{1}H and ^{13}C NMR spectra of a H_2S loaded aqueous solution of MDEA containing ethanol (EOH) and the corresponding spectra of the unloaded solution are shown in Figures 6 and 7 together with the peak assignment. Again, only peak shifts which are caused by the shift of the pH value are observed upon loading with H_2S, but no new peaks. Thus, a reaction of EOH or MDEA with H_2S can be excluded.

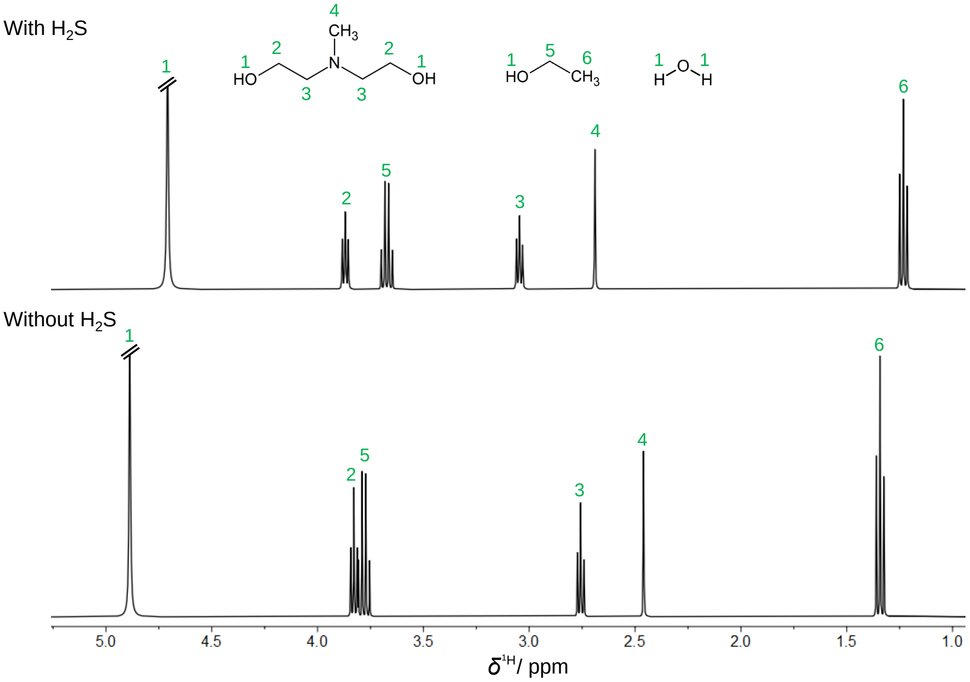

Figure 6: ^{1}H NMR spectra of a H_2S loaded aqueous solution of MDEA + EOH (top) and of the corresponding unloaded solution (bottom). Solvent composition, see Table 2, solvent saturated with H_2S at 323.15 K and 2.7 bar.

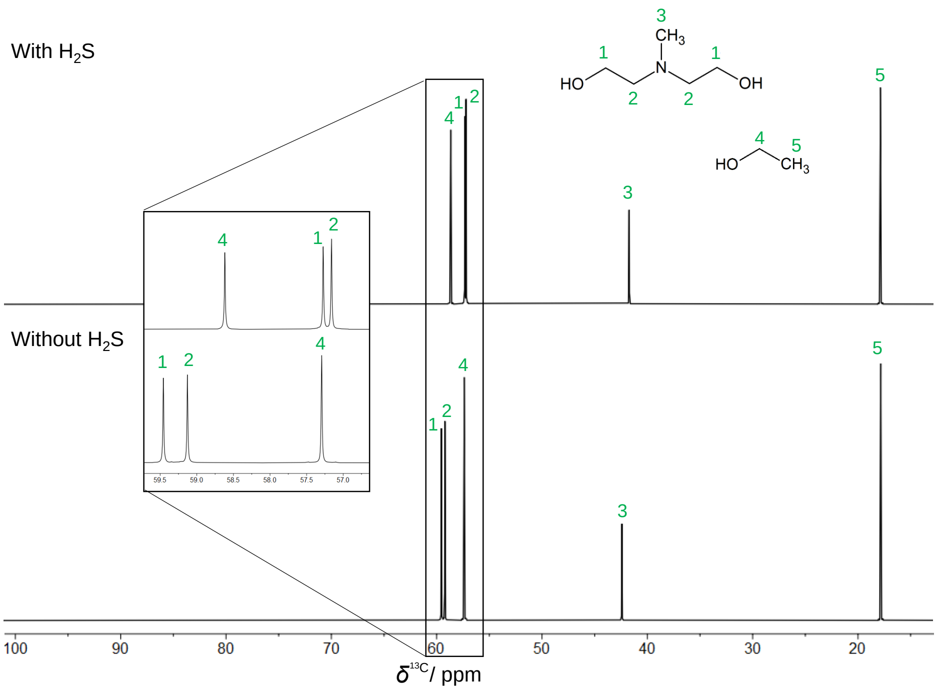

Figure 7: ^{13}C NMR spectra of a H_2S loaded aqueous solution of MDEA + EOH (top) and of the corresponding unloaded solution (bottom). Solvent composition, see Table 2, solvent saturated with H_2S at 323.15 K and 2.7 bar.

Conclusions

The speciation of solutions containing H_2S + MEA + W, H_2S + MDEA + PZ + W, and H_2S + MDEA + EOH + W was investigated in this study by NMR spectroscopy. A comparison of ^{1}H and ^{13}C NMR spectra of H_2S-loaded solutions and the unloaded solutions shows differences resulting from peak shifts that are induced by the shift of the pH value, but no signs of reaction products of H_2S with amines and alcohols. Hence, the assumption that only the dissociation of H_2S and water and the protonation of the solvent components needs to be taken into account in the modeling of the H_2S capture with aqueous amine solutions (including also alcohols as co-solvents) is clearly supported by the results from the present study.

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