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Journal of Sulfur Chemistry

Publication details, including instructions for authors and subscription information: http://www.tandfonline.com/loi/gsrp20

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Published online: 12 Aug 2015.

To cite this article: E. Avella-Moreno, N. Nuñez-Dallos, L. Garzón-Tovar & A. Duarte-Ruiz (2015): Reactions and products revealed by NMR spectra of deuterated dimethylsulfoxide with iodomethane in neutral and basic media, Journal of Sulfur Chemistry, DOI: 10.1080/17415993.2015.1066376

To link to this article: <u>http://dx.doi.org/10.1080/17415993.2015.1066376</u>

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Reactions and products revealed by NMR spectra of deuterated dimethylsulfoxide with iodomethane in neutral and basic media

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(Received 22 June 2015; accepted 23 June 2015)

Reactions occurring within each one of two mixtures, a mixture of deuterated dimethylsulfoxide, DMSO- d_6 , with CH₃I (system I) and another mixture of DMSO- d_6 with CH₃I, NaOH and water (system II), were monitored by 1D and 2D nuclear magnetic resonance (¹H, ¹³C, heteronuclear multiple quantum correlation, heteronuclear multiple bond correlation and diffusion-ordered NMR spectroscopy). The analysis of the spectra as a function of reaction time revealed the formation of methoxybis(trideuteromethyl)sulfonium iodide, **3**; the precipitation of hexadeuterated trimethyloxosulfonium, **2a**; a methyl exchange between DMSO- d_6 and **2a** to produce trideuterated dimethylsulfoxide, DMSO- d_3 , **4**, and nona-deuterated trimethyloxosulfonium iodide, **2b**; and the production of small quantities of methanol, **5**, trideuterated dimethylsulfide, **6**, and dimethyl ether, **7**, in both systems. Only system II precipitated deuterated [Na₄(DMSO- d_x)₁₅][(I₃)₃I], **1a**, a green solid with metallic shine that corresponds to an isotopomer of **1**, which is produced by the self-assembly of DMSO and CH₃I in the presence of NaOH and water.



Keywords: iodomethane; deuterated dimethylsulfoxide; NMR analysis of reaction mixtures; isotopomer of [Na₄(DMSO)₁₅][(I₃)₃I]; methyl exchange

1. Introduction

Dimethyl sulfoxide (DMSO), $CH_3(S=O)CH_3$, was obtained for the first time in 1867 by Alexander Mikhaylovich Zaytsev through the oxidation of dimethyl sulfide, CH_3SCH_3 . It is widely known as a polar aprotic solvent and for its usefulness in medicine.[1,2] DMSO is nucleophilic through oxygen (hard donor) or sulfur (soft donor), electrophilic in virtue of the weak acidity of its hydrogens, and it acts as an oxidizing or reducing agent with some compounds.[3,4] These qualities allow it to solvate hydrophilic and hydrophobic substances, to form complexes

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with metals and with organic substances, and to generate cations such as alkoxy dimethylsulfonium, $(CH_3)_2(^+S-OR)$, alkyl dimethyloxosulfonium, $(CH_3)_2R(^+S=O)$, and ylide anions $CH_3R(^+S=O)CH_2^-$, among other products.[3–11]

In nuclear magnetic resonance (NMR) spectroscopy, hexadeuterated dimethyl sulfoxide (DMSO- d_6), CD₃(S=O)CD₃ is a common solvent, and is not expected to react with solutes. It is used for its capacity to dissolve heterocyclic compounds and salts that are insoluble in other solvents, but its low volatility, its viscosity, its high affinity for water, and eventually some reactivity make it an inconvenient solvent.[11]

Recently the synthesis of $[Na_4(DMSO)_{15}][(I_3)_3I]$, **1**, was published. Compound **1** is a hexacoordinated sodium complex that is formed through a self-assembly process driven by ion–dipole interactions, where DMSO molecules are connected with Na⁺ ions to form a one-dimensional (1D) oligomeric structure stabilized by linear chains of triiodide (I_3^-) and iodide (I^-) anions along the crystal.[12]

In that earlier work, **1** was synthesized at room temperature in a reaction medium containing DMSO, iodomethane (CH₃I), sodium hydroxide (NaOH) and water in which trimethyloxosulfonium iodide was also obtained, [(CH₃)₃SO]I, **2**, [12] (some details of the structures of **1** and **2** are given in supplementary information). Since information published about the chemical reactions that occur in this reaction medium was scarce,[12,13–15] this work proposes to study two reaction systems (I and II) by NMR as a function of time in analogous conditions to those in the synthesis of **1** and **2**, with the objective of better understanding what happens during the process and determining the possible species and reactions that contribute to the formation and stabilizing of the oligomeric structure of **1**. System I is a mixture of DMSO- d_6 with CH₃I and system II is a mixture of DMSO- d_6 with CH₃I, NaOH and water. An analysis is presented for the evolution of the signals in a series of ¹H NMR and ¹³C NMR spectra of both systems which were maintained for more than 300 days in sealed tubes at 295 ± 2 K, through which the reactivity of DMSO- d_6 with CH₃I is observed.

2. Results and discussion

Prior to and during the monitoring of systems I and II containing CH₃I in DMSO- d_6 , the NMR spectra showed signals in addition to those expected if no reaction had occurred within the media. Those signals include a quintet at δ 2.50 (DMSO- d_5) and singlets at δ 2.18 (CH₃I), δ 3.33 or 3.30 (H₂O) and δ 3.53–3.57 (1,4-dioxane) in ¹H NMR spectra, and a septet at δ 39.5 (DMSO- d_6) and singlets at δ – 21.0 (CH₃I) and at δ 66.3 (1,4-dioxane) in ¹³C NMR spectra.[16–18]

Signals in the spectra of both systems provide evidence of a reaction occurring by showing consumption of CH_3I and production of other chemical species (Figures 1 and 2). 1,4-Dioxane was used as an internal standard for quantification. The water signals correspond to a residual signal in system I and the water added in system II.

The assignment of signals in ¹H NMR and ¹³C NMR spectra in systems I and II (Table 1) was confirmed through acquisition of two-dimensional NMR spectra (heteronuclear multiple quantum correlation (HMQC), heteronuclear multiple bond correlation (HMBC) and diffusion-ordered NMR spectroscopy (DOSY)) and compared using the shape and chemical shifts (δ) of signals with information in published spectra [12,16,17,19] or simulated ones using specialized software.[18]

In ¹H NMR spectra from both systems, there was a widening and decreasing of the intensity of the water signal and a persistence of the intensity of the 1,4-dioxane signal. A slight variation in the chemical shift, δ , was observed in some of the signals and was attributed to changes in composition of the environment in terms of nature and solute concentration due to reactions that slowly diversified the interactions between the species and modified the electromagnetic



Figure 1. ¹H NMR spectra of system I showing the variation of the signals intensities vs. time (d: days, h: hours after mixture of CH₃I with DMSO- d_6). **2a**, hexadeuterated trimethyloxosulfonium iodide; **3**, methoxy-bis(trideuteromethyl)sulfonium iodide; **4**, DMSO- d_3 ; **5**, methanol and **6**, CH₃SCD₃. Extension: δ 4.08–3.75 (left) and δ 2.60–2.45 (right).

environment of the nucleus to those that correspond to the signals.[11] The most notable δ variations in the signals of the system I spectra (Figure 1) were those of water (δ 3.29 \rightarrow 3.54), **2a** (δ 3.83 \rightarrow 3.91 \rightarrow 3.89), 1,4-dioxane (δ 3.57 \rightarrow 3.54) and CH₃I (δ 2.18 \rightarrow 2.13). In the system II spectra (Figure 2), there were small δ changes in water signals (δ 3.37 \rightarrow 3.39 \rightarrow 3.41 \rightarrow 3.53), **2a** (δ 3.82 \rightarrow 3.91 \rightarrow 3.90), 1,4-dioxane (δ 3.55 \rightarrow 3.53) and CH₃I (δ 2.16 \rightarrow 2.13).

The concentration of species evolved during the reaction was determined by the integration of signals in the ¹H NMR spectra series of systems I and II and was normalized with respect to maximum concentration, as shown in Figures 3 and 4. The changes in intensity of the signals in the ¹H NMR spectra (Figures 1 and 2) and those in the concentration of the respective species were evidence of the consumption of CH₃I from the beginning of reaction and until its depletion in both systems. Initially, a signal with very low intensity in δ 3.99 (¹H) and in δ 62.1¹ (¹³C), attributable to methoxy-bis(trideuteromethyl)sulfonium iodide 3, appeared, and was rapidly extinguished, while another signal that was attributed to **2a**, appeared in δ 3.89 (¹H) and δ 39.2 (¹³C), as illustrated in Scheme 1. These results agree with those published by Smith and Winstein about isomerization in solution, promoted by the nucleophilic character of the nonbonding pairs of electrons in the sulfur atom of the alkoxy dimethylsulfonium cation to produce an alkyl dimethyloxosulfonium cation. [15] However, it does not exclude that 3 could be an isomeric contact ion pair of 2. 2a is detected by NMR, without isolating it from the medium and by producing it in a closed recipient, without inert atmosphere, refluxing or anhydrous DMSO, under different experimental conditions and having reagents in different proportions to those used by Kuhn and Trischmann [13,14] or other authors, to obtain 2 or some isotopomer of 2.[20-24]

After the concentration of **2a** reached a maximum point, it diminished while the trideuterated dimethylsulfoxide, DMSO- d_3 (4) signal appeared (¹H NMR: δ 2.54 and ¹³C NMR: δ 40.3). This behavior is attributed to the exchange of CH₃ for CD₃ between **2a** and DMSO- d_6 described in Scheme 2, to produce DMSO- d_3 and nona-deuterated trimethyloxosulfonium iodide, 2b,



Figure 2. ¹H NMR spectra of system II showing the variation of the signals intensities vs. time (d: days, h: hours after mixture of CH₃I, DMSO- d_6 , NaOH and water). **2a**, hexadeuterated trimethyloxosulfonium iodide; **3**, methoxy-bis(trideuteromethyl)sulfonium iodide; **4**, DMSO- d_3 ; **5**, methanol; **6**, CH₃SCD₃ and **7**, dimethyl ether. Extension: δ 4.04–3.78 (left) and δ 2.58–2.46 (right).

(¹³C NMR: δ 38.4). In both systems, there was an increase in the concentration of **4** while **2a** and CH₃I diminished. It was determined that the exchange of CH₃ for CD₃ happened between **2a** and DMSO-*d*₆ because the concentration of **4** increased during the rest of the monitoring, even after CH₃I was depleted, and from the remaining species in the medium, **2a** was the only one whose concentration continued diminishing (Figures 3 and 4). As an antecedent of obtaining products from this exchange, Ripmeester published that by decomposition at 190°C and in a vacuum, a solution of **4** in DMSO-*d*₆ was produced from **2a**.[20]

As time went on in systems I and II, the red color of the medium was intensified due to the generation of iodine, I_2 , and, at the same time, precipitation of a white solid that we attribute to the isotopomers from **2**, **2a** and **2b**, given the similarity of the appearance of the solid and the chemical shift of the signals observed in the spectra with those from **2**, as recorded in a previous study.[12] The start of the precipitation of the white solid coincided with reaching the maximum recorded concentration of **2a** (at 11.9 days, in system I, and at seven days in system II). Only in system II was a compound which was solid green with metallic shine precipitated, which is attributed to deuterated Na₄(DMSO- d_x)₁₅][(I₃)₃I], **1a**, an isotopomer of **1** whose physical appearance and the chemical shift of the signals in the spectra are very similar to those of product **1** that was obtained in similar conditions by self-assembly between DMSO and CH₃I.[12] The start of precipitation of **1a** also coincided with the maximum recorded concentration of **2a** and **1a** is preceded by a saturation of the solution of the medium with **2a**.

In both systems, the formation of methanol, CH₃OH, **5**, (¹H, δ 3.13 and ¹³C, δ 48.5), CH₃SCD₃, **6**, (¹H, δ 2.92 and ¹³C, δ 25.9) and dimethyl ether, **7**, CH₃OCH₃ (¹H, δ 3.16 and

¹ H NMR		¹³ C NMR		
δ:	Multiplicity	δ:	Multiplicity	Assignment
2.18 ^a	S	-21.44^{a}	S	CH ₃ (CH ₃ I)
2.5	Quintet	39.51	Septet	CHD_2 (DMSO- d_5) and CD_3 (DMSO- d_6)
2.52	m	39.77	\hat{M}	CHD ₂ and CD ₃ ([Na ₄ (DMSO- $d_x)_{15}$][(I ₃) ₃ I], 1a)
2.54	S	40.29	S	CH_3 (DMSO- d_3 , 4)
2.92 ^a	S	25.87 ^a	S	CH_3 (dimethylsulfide, 6)
3.13 ^a	S	48.53 ^a	S	CH ₃ (CH ₃ OH, 5)
3.16 ^a	S	59.77 ^a	S	CH_3 (dimethyl ether, 7)
3.29 ^a	s (broad)			H (H ₂ O)
3.57 ^a	S	66.29 ^a	S	CH ₂ (1,4-dioxane)
3.89 ^a	S	39.18 ^a	S	CH ₃ S (hexadeuterated trimethyloxosulfonium iodide, 2a)
3.99 ^a	S	62.11 ^{a,b}	S	CH ₃ O (methoxy-bis(trideuteromethyl)sulfonium iodide, 3)

Table 1. The assignment of the signals of ¹H NMR and ¹³C NMR spectra in systems I (DMSO- d_6 and CH₃I) and II (DMSO- d_6 , CH₃I, NaOH and H₂O).

Note: *m*, multiplet, *s*, singlet.

^aChemical shift of these signals changed slightly during the process. That was attributed to effects of changes in concentration and interactions between substances in the reaction mixture.

^bIt is a low-intensity signal attributable to CH₃ in **3**. The small septet expected for the CD₃ in **3**, apparently is overlapped with the big signal of DMSO- d_6 (\approx 39.51 ppm).



Scheme 1. Formation of 3 and 2a from CH₃I and DMSO-d₆.



Scheme 2. Exchange of CH₃ for CD₃ between 2a and DMSO-d₆.

¹³C, δ 59.8) was detected. For comparable times measured from the beginning of the process and in relation to the amount of CH₃I initially allowed, system II produced less quantity of **2a** and DMSO-*d*₃, but generated a little more of **5**, **6** and **7** than in system I. These differences were attributed to the addition of NaOH and water in system II, which compete with DMSO-*d*₆ for the CH₃I in the substitution in which **2a** is formed to produce methanol, **5**, and dimethyl ether, **7**.

For that reason, the maximum concentration of **2a** in system II was less that in system I and was reached 4 days before (Figures 3 and 4). On the other hand, in the ¹H NMR spectra of system II, signals with very low intensity were observed, corresponding to hydrogen bonded to carbon sp³, characteristic of aliphatic compounds, attributable to ethane (δ 0.90–0.95) and to other hydrocarbons present in such scarce concentration that they could not be quantified with certainty.

The results of NMR monitoring allowed for proposing the sequence of reactions shown in Scheme 3 to explain the formation of **1a** in system II as an oligomeric structure stabilized by triiodide anions (I_3^-) and iodide (I^-) obtained by self-assembly in solution between DMSO- d_6 and CH₃I, in which production of trideuterated dimethyl sulfide **6**, is evidenced, by the reduction





Figure 3. Evolution of the concentration of four major components of system I.



Figure 4. Evolution of the concentration of six major components of system II.

of DMSO- d_3 , **4**, with HI,[25] together with ethane, and iodine, generated probably by photolysis of CH₃I,[26] among other detected products (2a, 5, 7). In order to complement our work we are considering to study the reaction between DMSO and CH3I with small additions of aqueous HI.

The ¹H NMR and ¹³C NMR DOSY spectra of system II, acquired toward the end of the monitoring period, evidenced that signals assigned to different chemical species in the NMR spectra effectively correspond to different discrete entities present in the mixture. Up to six different diffusion coefficients were found that correlate to the chemical shift of DMSO-*d*₃, **4**, methanol,

CH₃I + NaOH CH₃OH + NaI 5 CH₃OH + NaOH CH₃ONa + H₂O 5 CH₃ONa + CH₃I CH₃OCH₃ + NaI 7 $CH_{3}I + H_{2}O$ CH₃OH + HI 5 $CH_3(S=O)CD_3 + 2 HI$ $CH_3SCD_3 + H_2O + I_2$ 4 hv 2 CH₃I CH₃CH₃ + I₂ NaI + I₂ NaI3 ____ Self-assembly process $15 \text{ DMSO-} d_6 + 3 \text{ NaI}_3 + \text{NaI}$ [Na4(DMSO-d6)15][(I3)3I] 1a

Scheme 3. Sequence of chemical reactions in system II that explains the formation of 1a.

5, trideuterated dimethyl sulfide, 6, dimethyl ether, 7, hexadeuterated trimethyloxosulfonium, 2a, water, 1,4-dioxane, DMSO- d_5 and DMSO- d_6 .

In ¹³C NMR spectra of systems I and II, acquired at the end of monitoring period, signals were observed that confirm the predominance of DMSO- d_3 , **4**, and the existence of small quantities of **2a**, and additional low-intensity signals were detected that gave indication of the formation of deuterated species corresponding to deuterated methanol, **5b**, at δ 47.6, hexadeuterated dimethyl sulfide, **6b**, at δ 25.5, and hexadeuterated dimethyl ether, **7b**, at δ 59.6, as products of the chemical exchange of hydrogen or methyl between species present in the medium. However, this information is not enough to establish with certainty the mechanism by which this exchange occurs.

3. Conclusions

Monitoring of a mixture of DMSO- d_6 with CH₃I (system I) and of another mixture of DMSO d_6 with CH₃I, NaOH and water (system II) by means of NMR analysis, at 295 \pm 1 K for more than 300 days allowed detection of chemical species that are formed, and the propounding of concurrent chemical reactions for the production of $[Na_4(DMSO)_{15}][(I_3)_3I]$, 1. In both systems, there was a consumption of CH₃I and DMSO- d_6 to produce 3 (methoxy-bis(trideuteromethyl)) sulfonium iodide) and indications of the fast isomerization of the cation of 3 to produce 2a (hexadeuterated trimethyloxosulfonium iodide) was seen. An exchange of CH₃ by CD₃ was detected between 2a and DMSO- d_6 to produce 4 (DMSO- d_3) and 2b (nona-deuterated trimethyloxosulfonium iodide) that occurred later, at room temperature (295 \pm 2 K) and local pressure $(\cong 563 \text{ mm Hg})$. 2a, the white solid that precipitated from the reaction medium, and 2b were attributed to isotopomers of 2 (trimethyloxosulfonium iodide). Additionally, in both systems small quantities of methanol, 5, trideuterated dimethyl sulfide, 6, and dimethyl ether, 7, were detected. Only in system II, **1a** ([Na₄(DMSO- $d_x)_{15}][(I_3)_3I]$ deuterated) was obtained as a green precipitate very similar to 1, and a greater quantity of 5, 6, and 7 was detected than in system I. This is attributed to the presence of NaOH and water from the beginning in the mixture of system II.

The results obtained during this monitoring enabled the proposal of a sequence of reactions to explain the formation of **1a** in system II and prove that there is considerable reactivity of DMSO- d_6 with CH₃I when acquiring and analyzing NMR spectra, obtained in this solvent.

4. Experimental

4.1. General

The reagents, DMSO (Carlo Erba, 99.9%), DMSO- d_6 (Sigma-Aldrich, 99.9%-D), CH₃I (Merck, 98%, stabilized with Ag), NaOH (Merck, \geq 97%, pellets), distilled water and 1,4-dioxane (Sigma-Aldrich, 99.8%) were used without additional purification. 1,4-dioxane was used as an internal standard for quantification.

4.2. Preparation of Systems I and II

The solutions for Systems I and II were prepared by weighing the components on an analytic scale, directly within the NMR tube (Norell[®], Item No. 507-HP-8; 5 mm OD and 8 inch).

System I: 1.0552 g of DMSO- d_6 (in excess), 0.2121 g of CH₃I and 0.0102 g of 1,4-dioxane.

System II: 1.0398 g of DMSO- d_6 (in excess), 0.2135 g of CH₃I, 0.0105 g of NaOH, 0.0081 g of distilled H₂O and 0.0124 g of 1,4-dioxane.

The solutions in the tubes were frozen with liquid nitrogen, and the tubes were sealed at once with an oxyacetylene flame at atmospheric pressure (563 mmHg). Later they were kept at 295 ± 2 K (laboratory temperature). In that way, they were undisturbed most of the time; they were only agitated mechanically (DAIGGER Vortex Genie 2, Cat. No. 222204) for 3 min before each NMR spectra acquisition.

4.3. NMR

The NMR spectra of each system were acquired at 295 ± 1 K in a Bruker Avance 400 spectrometer outfitted with a broadband observe probe, operating at 400.13 MHz for ¹H and at 100.62 MHz for ¹³C. The residual DMSO signal was used as a scale reference in the ¹H NMR (δ : 2.50, quintet, ²J_{HD} \cong 1.9 Hz) and ¹³C NMR (δ : 39.5, ¹J_{CD} \cong 21.0 Hz) spectra. The integration of the 1,4-dioxane (δ : 3.53–3.57) in ¹H NMR [13,14] signal was used as an internal pattern for quantification, following prescriptions given in the literature.[27–29] Each FID was processed 10 times until obtaining integral averages with variation coefficients (CV) \leq 5%, in intervals with 95% confidence limits (according to test *t* from Student) for signals with signal to noise at \geq 150:1 (MestReC[®]).[30]

The ¹H NMR and ¹³C NMR spectra were acquired for system I from 0.3 h to 343 days (d) 0.4 h after the mixture of reagents, and for system II from 1.8 h to 307 d 0.3 h. The assignment of the signals, as seen in Table 1, and the composition of the reagent mixtures was verified by means of the acquisition of 2D NMR spectra (HMQC, HMBC and DOSY). The acquisition parameters of all the NMR experiments, other NMR (¹³C, HMQC, HMBC and DOSY) spectra, and some useful enlargements of these are available in the supplementary information.

Disclosure statement

No potential conflict of interest was reported by the authors.

Supplemental data

Supplemental data for this article can be accessed at http://dx/doi.org/10.1080/17415993.2015.1066376.

Note

1. It is a low-intensity signal attributable to CH₃ in **3**. The small septet expected for the CD₃ in **3**, apparently is overlapped with the big signal of DMSO- d_6 (\approx 39.51 ppm).

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