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TECH NOTE: 22-001

Handling of NMR Solvents

Abstract

This tech note describes the proper way to handle NMR solvents available from Zeotope. The use of inappropriate materials for the sampling the NMR solvents can lead to the introduction of impurities that are not otherwise present in the high purity solvents. These impurities may then be observed in NMR spectra acquired using these solvents.

Glass vs Plastic

The use of plastic pipettes or syringes to transfer NMR solvents can easily introduce impurities, observable via ^{1}H-NMR spectroscopy, into the sample. For this reason, Zeochem recommends the use of glass pipettes or glass syringes equipped with stainless steel cannulas for the handling of our high purity solvents.

The most commonly used plastics for disposable labware are PP and PE. The caps for NMR tubes may be made from PP, so impurities may be seen if the solvent comes into contact with the cap, for instance via shaking. Plastics can contain many different types of additives: antioxidants, lubricants, plasticizers, release agents, and slip agents.(1) The release agents are a particularly likely source of impurities, they are used to ensure the plastic can be easily removed from the mold and are therefore likely to be found on the surface. Common release agents include metal stearates and polydimethylsiloxane, shown in Figure 1. Common lubricants include fatty alcohols and fatty acid esters with carbon chain lengths of 14 to 18. The methyl groups at the end of the chains are observed in the region δ = 0.6-2.0 ppm in ^{1}H-NMR spectra, while the methylene groups in the chains are observed in the region δ = 1.2-2.3 ppm in ^{1}H-NMR spectra.(2) The methyl groups of silicone greases, such as polydimethylsiloxane, are observed in the region δ = –0.1-0.3 ppm in ^{1}H-NMR spectra.(3) This is not an exhaustive list of potential impurities and signals may be observed in other regions as well. These types of impurities are not commonly found on glass surfaces.

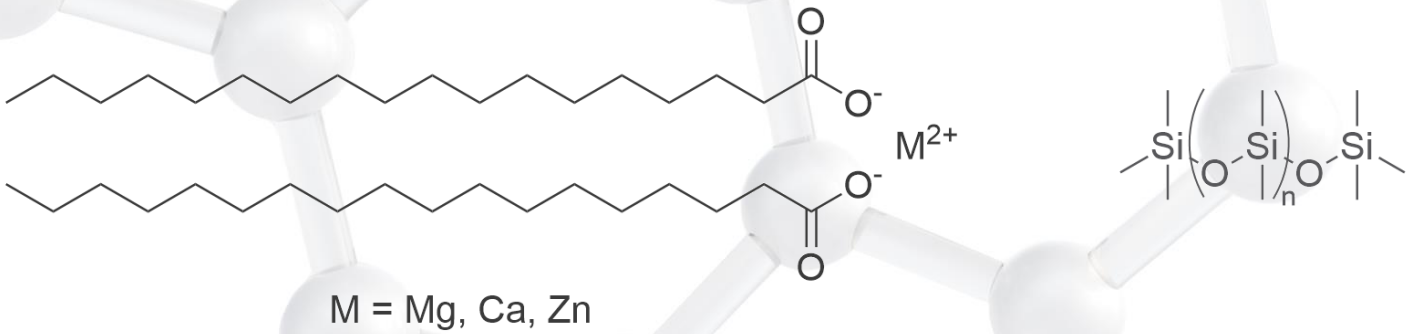

Figure 1: Structures of metal stearates (left) and polydimethylsiloxane (right).

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Examples

Some example spectra are given showing the impurities observed when the sample is taken using a plastic syringe.

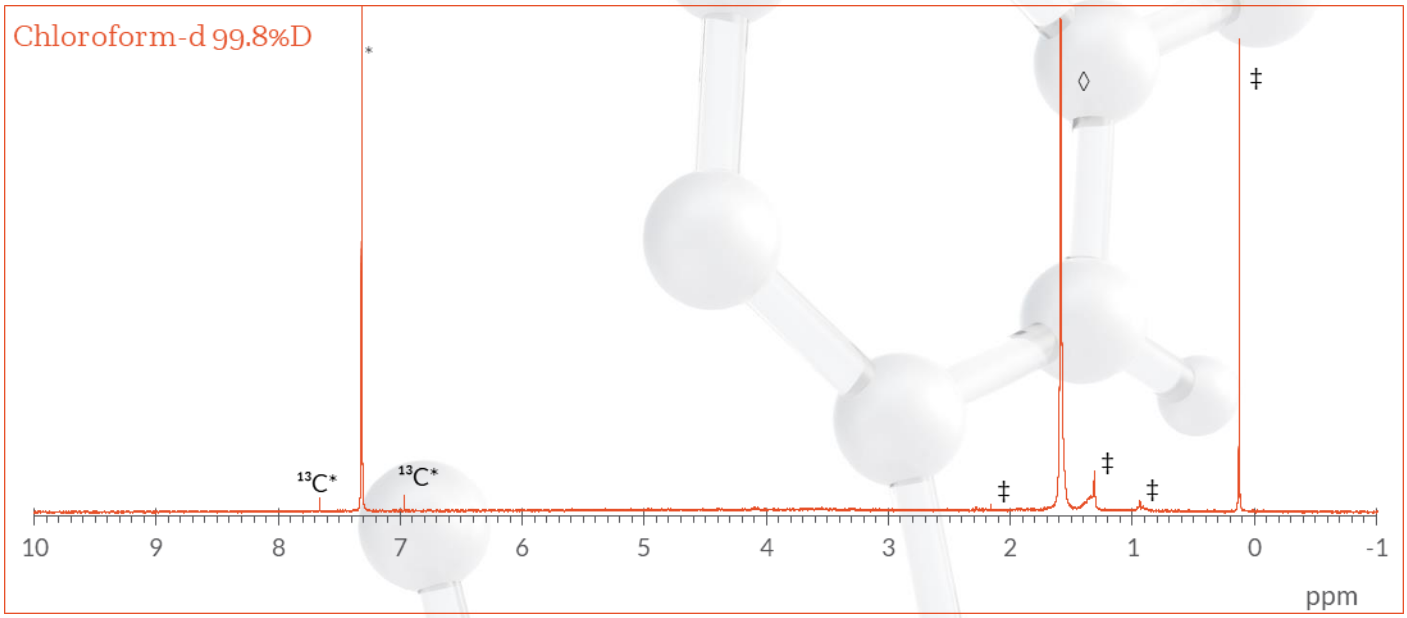

Figure 2: ^{1}H-NMR spectrum of Chloroform-d. Showing: * – residual solvent peak, $^{13}C^*$ – ^{13}C satellites of the residual solvent peak, ◊ – residual water peak, ‡ – impurities from the plastic syringe.

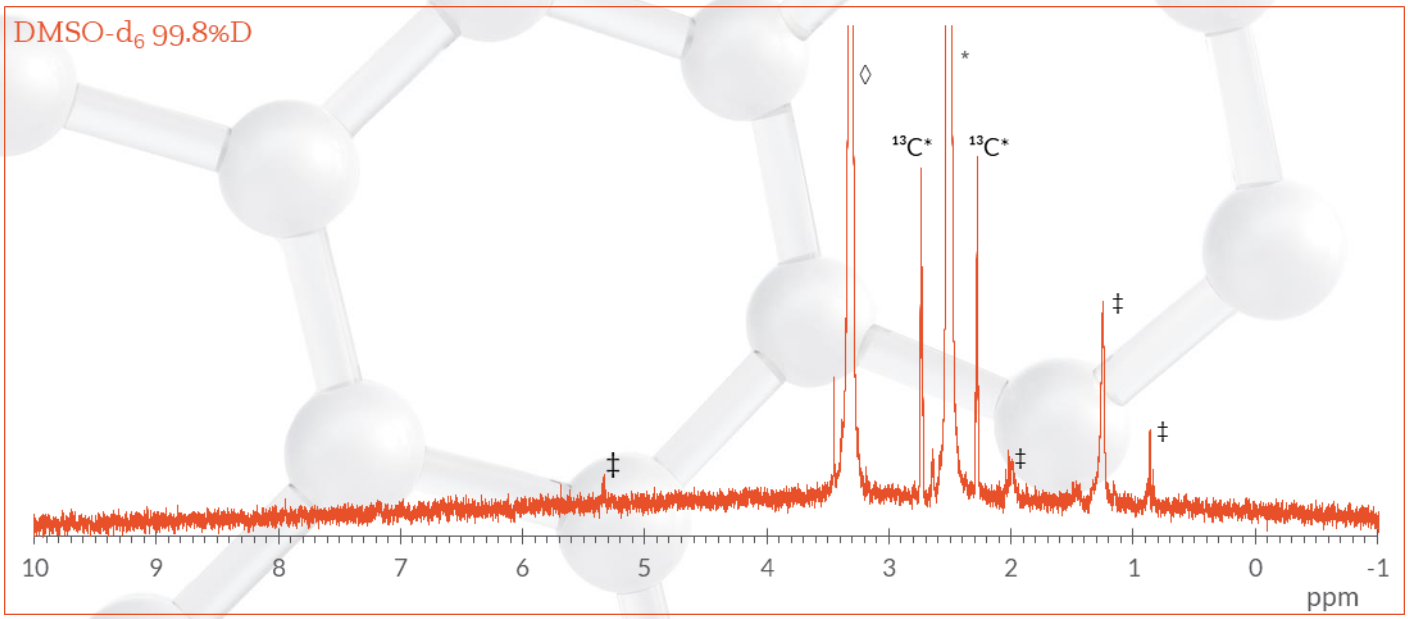

Figure 3: ^{1}H-NMR spectrum of DMSO-d_6. Showing: * – residual solvent peak, $^{13}C^*$ – ^{13}C satellites of the residual solvent peak, ◊ – residual water peak, ‡ – impurities from the plastic syringe.

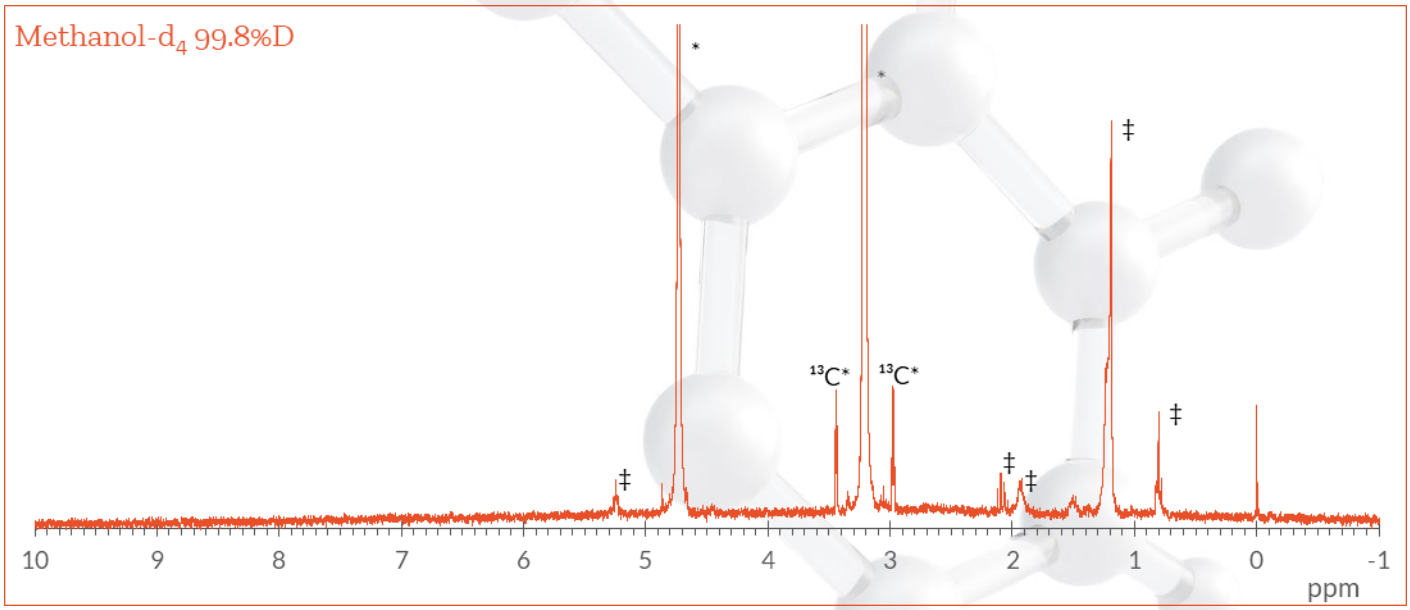

Figure 4: ^{1}H-NMR spectrum of Methanol-d$_4$. Showing: * – residual solvent peak, ^{13}C* – ^{13}C satellites of the residual solvent peak, ‡ – impurities from the plastic syringe.

References

(1) M. Bolger, J. Hubball, J. Groeger, S. Meronek, Handbook for the Chemical Analysis of Plastic and Polymer Additives, 2nd Edition, CRC Press, 2016

(2) E. Pretsch, P. Bühlmann, C. Affolter, M. Baderstcher, Spektroskopische Daten zur Strukuraufklärung organischer Verbindungen, Springer, 2001

(3) G. R. Fulmer et al., Organometallics, 2010, *29*, 2176-2179