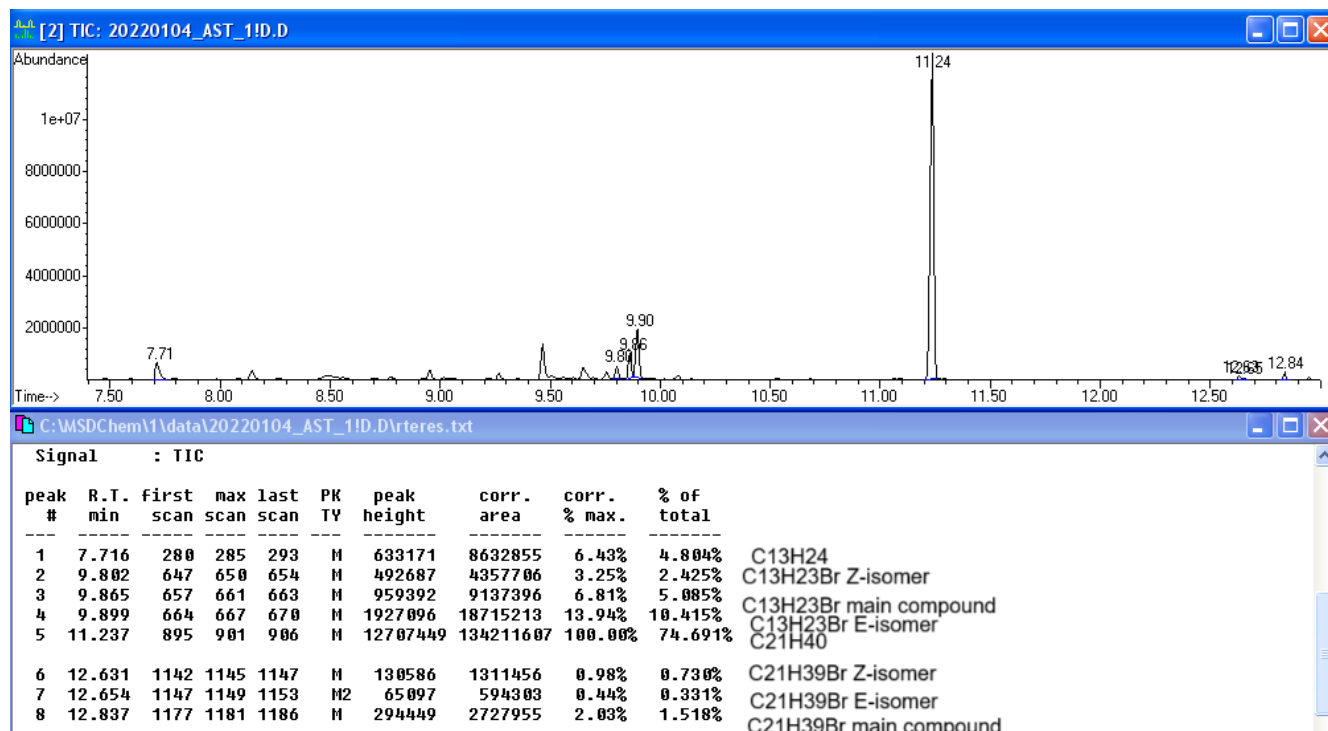
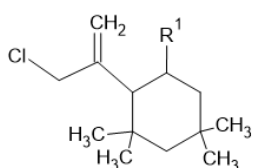


Which analytical standards are required for the characterization of halobutyl rubber?

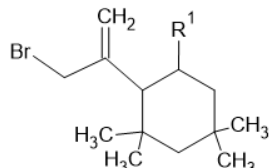
Rubber oligomers are common extractables and leachables from bromo/chlorobutyl rubber stoppers. A typical GC-MS chromatogram of a commercial bromobutyl rubber stopper is given below. The highest peak is rubber oligomer C₂₁H₄₀, followed by C₁₃H₂₃Br-E-isomer and C₁₃H₂₃Br main compound.



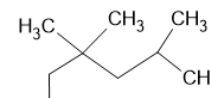
Nowadays, the 6 most common rubber oligomers are available as analytical standards: C₁₃H₂₄ (CAS 63251-38-7), C₂₁H₄₀ (CAS 2512216-71-4), C₁₃H₂₃Br (CAS 2514965-51-4), C₂₁H₃₉Br (CAS 2518227-14-8), C₁₃H₂₃Cl (CAS 63216-72-8) and C₂₁H₃₉Cl (CAS 2446375-29-5). The structure of the halogenated oligomers is given below:



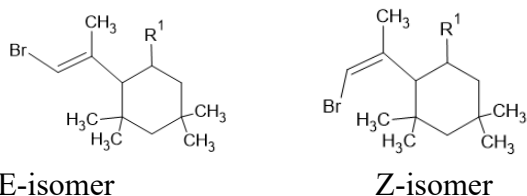
For C₁₃, R₁=H



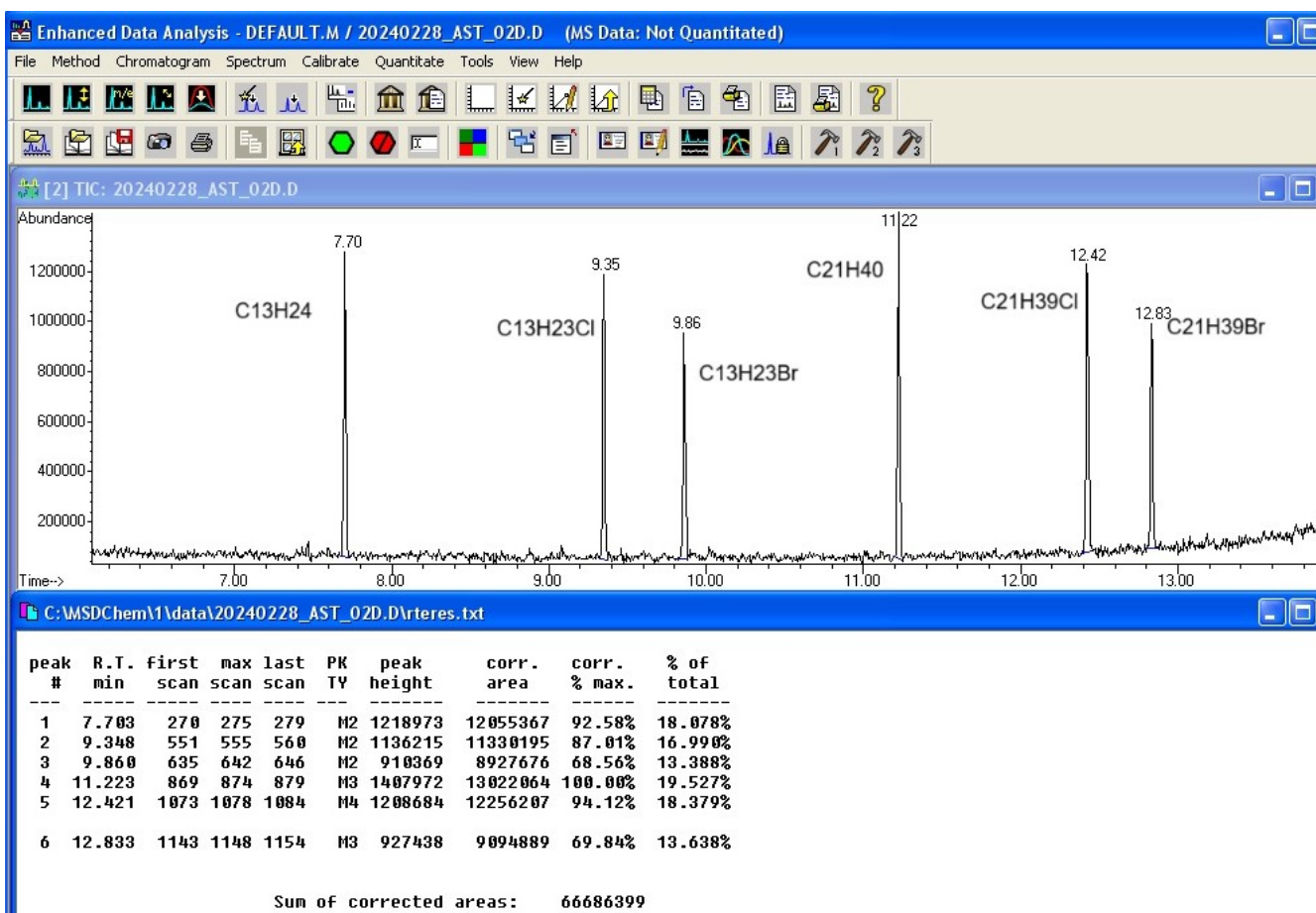
For C₂₁, R₁=



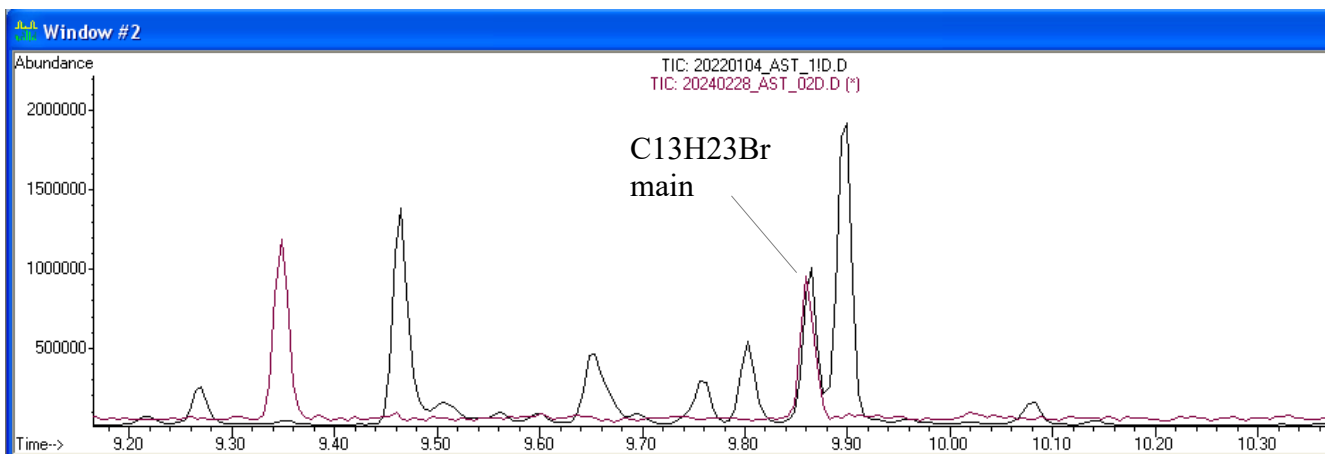
However, isomers of the halogenated oligomers exist, which (depended on the rubber formulation) can occur in higher concentrations than the “main” compound, as shown above for C13H23Br. The structure of these isomers has been elucidated, the results are available on [Researchgate](#) (example of C21H39Br isomers).



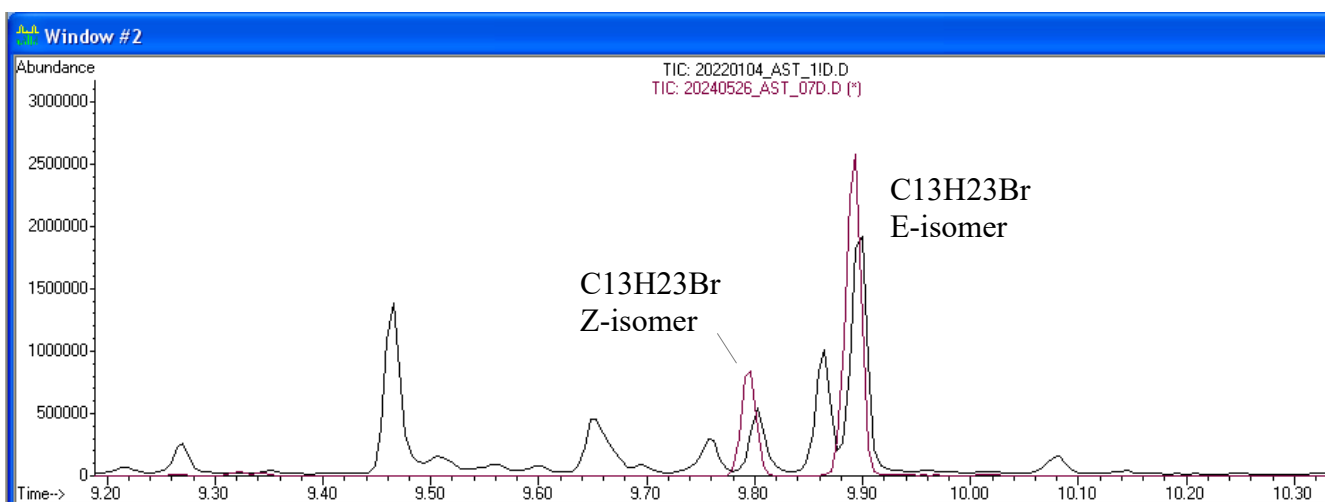
Below, a mix of the 6 common rubber oligomer standards is depicted.



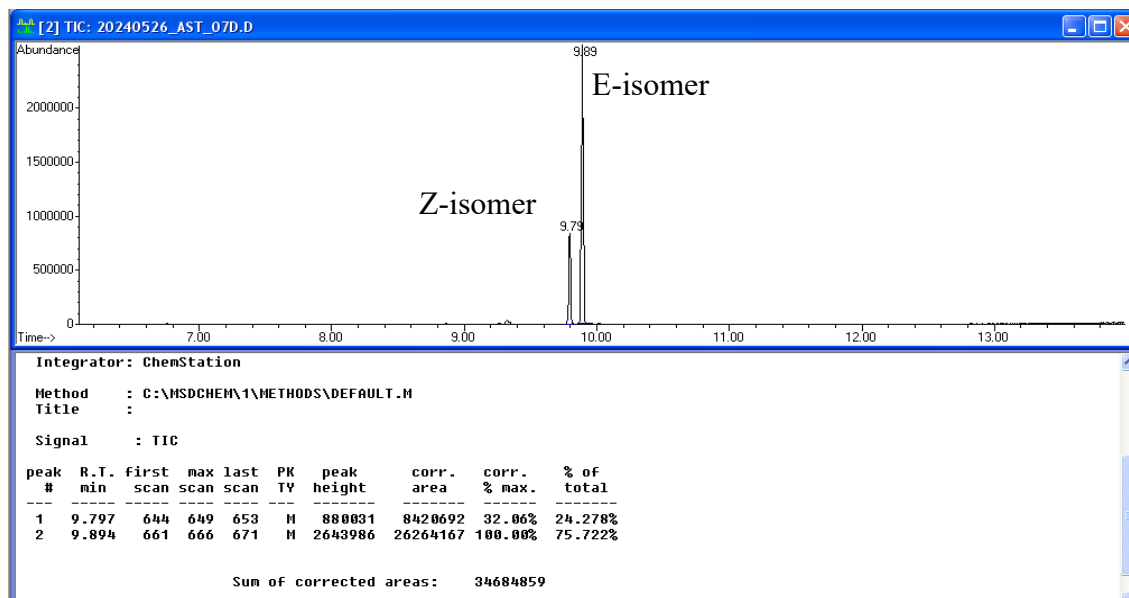
An overlay of the 6 common rubber oligomer mix and the bromobutyl rubber extract (C13H23Br region zoomed) clearly shows, that only a minor part of the C13H23Br oligomers is covered by the STD mix. By using a standard mix of C13H23Br Z- and E-isomers in addition, the peak group of C13H23Br can be completely identified and quantified. The same applies to C21H39Br the isomers.



red: 6 oligomer STD mix black: bromobutyl rubber extract

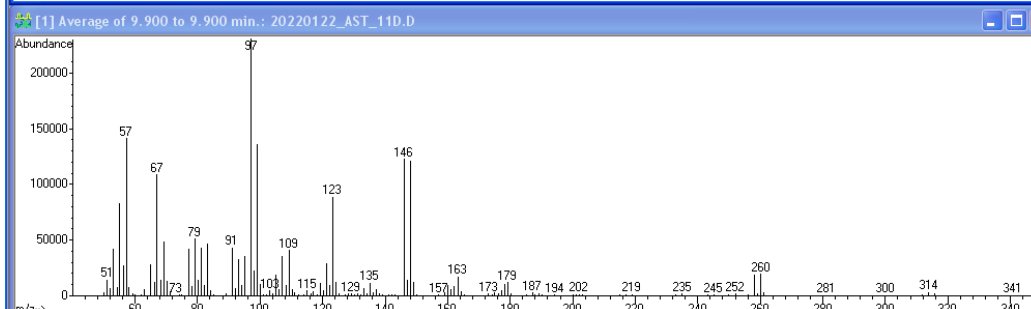
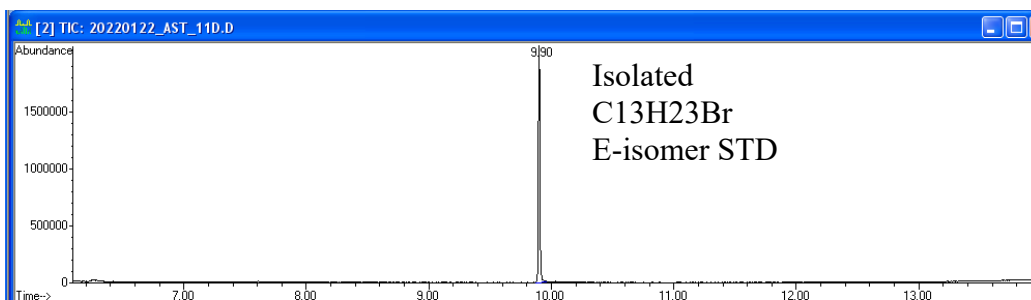
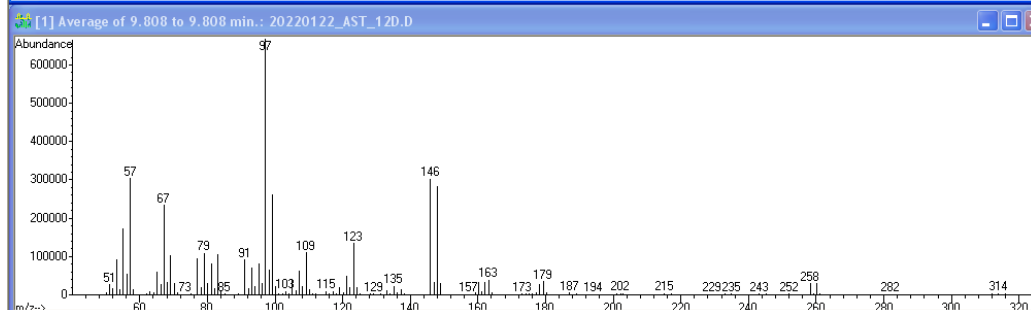
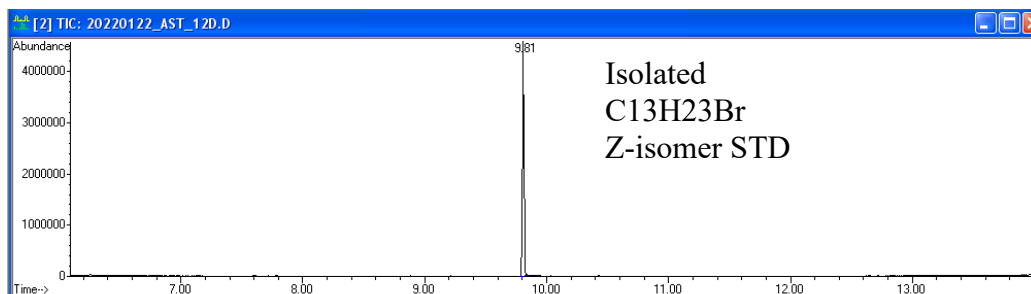


red: C13H23Br E/Z isomer STD mix black: bromobutyl rubber extract



Standard mix of C13H23Br Z- and E-isomers

The separation of the Z- from the E-isomer is also possible, but the procedure is complicated and costly.



I would therefore like to ask the E&L experts:

Do you see the need to use standards for rubber oligomer isomers as well?

If so, do you prefer to use a mixture (content of both compounds determined by quan-NMR of the mix), or do you prefer to use the individual, isolated compounds.

Thank you in advance for your answers and comments.