Chemical shift – theory

Ziel: structure determination of chemical substances

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- electron orbitals contribute to B_0:

$$\delta\vec{B} = \sigma\vec{B_0}$$

- modification of the Larmor frequency:

$$\omega_i = \omega_L\,(1 - \sigma_i)$$

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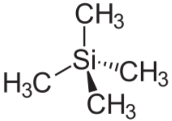
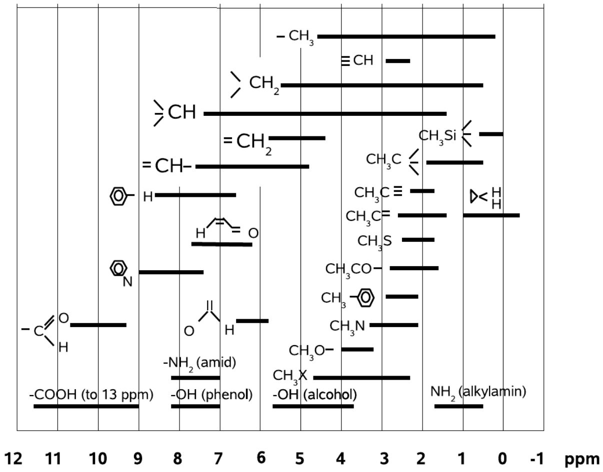
- reference substance: TMS (Tetra-Methyl-Silan)



- relative chemical shift in ppm:

$$\delta_i = \sigma_i - \sigma_{TMS} = \frac{\omega_{TMS} - \omega_i}{\omega_L}$$

Chemical shifts δ_i of compounds relative to TMS



Measurements

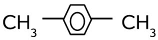
- five chemical substances, with and without TMS
- inhomogeneities and diffusion processes reduce resolution
  $\Rightarrow$ thin glass tube, put into rotation with pressure air
- result:
  - without rotation:
    FWHM = 200 Hz, I = 0,25
  - with rotation:
    FWHM = 20 Hz, I = 1,9
- energy resolution:
  $\Delta E_{NMR} = h \cdot \Delta\nu = 8,28 \cdot 10^{-14}\,\mathrm{eV}$

Idenfication of the Probes

Probe C: acetic acid $CH_3 - COOH$

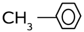
Peaks of C+ [ppm]	Peaks of C [ppm]	Chem. shift.	
$p_1 = 16,7$	$p_1 = 17,0$	$\delta_i = 11,6$	COOH-group
$p_2 = 26,2$	$p_2 = 26,6$	$\delta_i = 2,1$	Methyl group CH_3
$p_3 = 28,3$	–	–	TMS

Idenfication of the Probes

Probe B: p-Xylol 

Peaks of B+ [ppm]	Peaks of B [ppm]	Chem. shift	
$p_1 = 22,7$	$p_1 = 22,7$	$\delta_i = 7,0$	Benzene group
$p_2 = 27,5$	$p_2 = 27,5$	$\delta_i = 2,2$	Methyl group, Peak twice as high as p_1
$p_3 = 29,7$	–	–	TMS

Idenfication of the Probes

Probe E: toluol 

Peaks of E+ [ppm]	Peaks of E [ppm]	Chem. shift	
$p_1 = 19,5$	$p_1 = 23,1$	$\delta_i = 7,3$	Benzene group
$p_2 = 24,4$	$p_2 = 23,1$	$\delta_i = 2,4$	Methyl group, peaks have same hight
$p_3 = 26,8$	–	–	TMS

Idenfication of the Probes

Probe A: fluoroaceton $FCH_2 — CO — CH_3$

Peaks of A+ [ppm]	Peaks of A [ppm]	Chem. shift	
$p_1 = 22,2$	$p_1 = 23,8$	$\delta_i = 6,3$	FCH_2-group
$p_2 = 24,6$	$p_2 = 21,4$	$\delta_i = 3,9$	
$p_3 = 26,4$	$p_3 = 19,6$	$\delta_i = 2,1$	Methyl group CH_3
$p_4 = 28,5$	–	–	TMS

Idenfication of the Probes

Probe D: fluoroacetonitril FCH_2-CN

Peaks of D+ [ppm]	Peaks of D [ppm]	Chem. shift	
$p_1 = 30,8$	–	–	TMS
$p_2 = 34,8$	$p_2 = 26,6$	$\delta_i = 6,4$	FCH_2 group
$p_3 = 37,2$	$p_3 = 24,2$	$delta_i = 4,0$	

one dimensional imaging – theory

- **position dependent magnet fields**
- Superposition of the static field $\vec{B_0}$ with gradient fields $\vec{B^x}$, $\vec{B^y}$, $\vec{B^z}$
- two techniques:

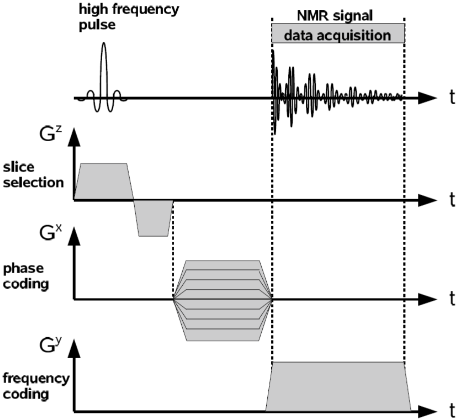
frequency coding

- Larmor frequency $\omega_L = \gamma(B_0 + G^z \cdot z) = \omega_L^0 + \omega_z$
- measured NMR signal $S(t)$ is Fourier transform of $M_\perp^{rot}(z)$

phase coding

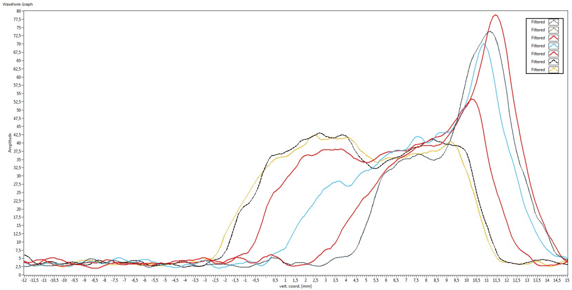
- apply gradient field, increase strength
- phase rotates: $\phi(z) = (\gamma G^z T_{Ph})z = k_z z$

two dimensional imaging – theory



One dimensional imaging measurements

- Bruker® NMR analyzer mq7.5
- Glass tube filled with 15 mm of oil
- Glass tube filled with 50 mm of water
- glass tube with teflon structure
- examination of an inflitration process:
  Fick's second law: $\frac{\partial c}{\partial t} = D\frac{\partial^2 c}{\partial x^2}$



two dimensional imaging measurements

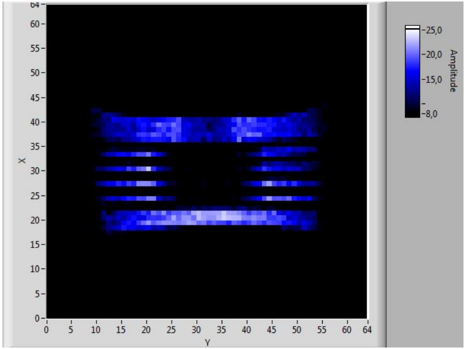


Figure: teflon structure

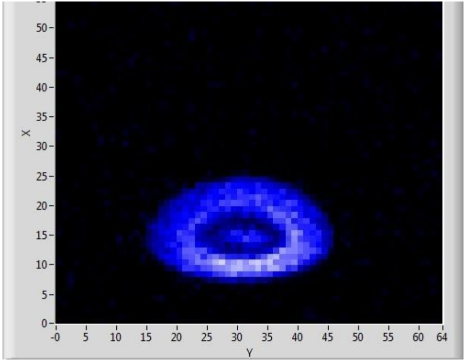


Figure: olive

two dimensional imaging measurements

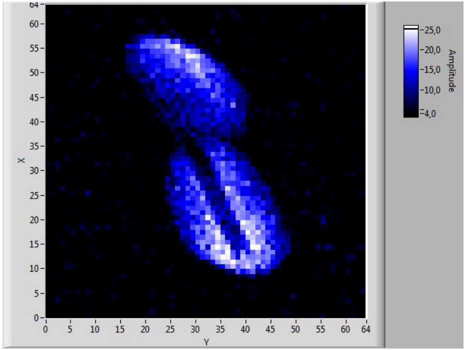


Figure: peanut shell

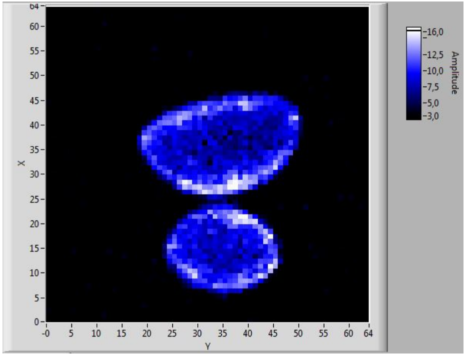


Figure: aloe vera