

Determining Molar Enthalpy and Entropy of Vaporization for Acetone and n-Hexane

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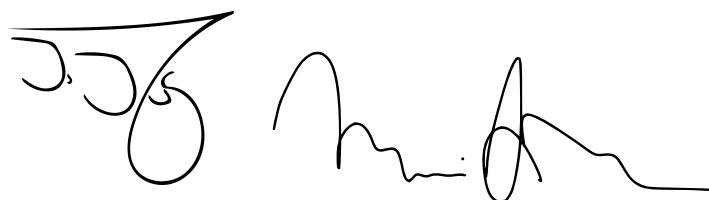
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Abstract

The molar enthalpy and entropy of evaporation of acetone and n-hexane were determined via two experiments. First, by measuring vapor pressure and boiling temperature between 100 mbar to 1000 mbar and 15 °C to 55 °C. By plotting the logarithmic pressure against the inverse temperature, thus linearizing the correlation, the molar enthalpy of evaporation $\Delta_V H$ was found using the Clausius-Clapeyron equation. Acetone was found to have $\Delta_V H = 32.49 \pm 0.26 \text{ kJ/mol}$ and n-hexane $\Delta_V H = 32.66 \pm 0.25 \text{ kJ/mol}$. With the Gibbs-Helmholtz equation, entropies of evaporation of $\Delta_V S(T_0) = 98.62 \text{ J}/(\text{mol K})$ for acetone and $\Delta_V S(T_0) = 95.74 \text{ J}/(\text{mol K})$ for n-hexane were determined for standard boiling temperature at 1 atm. In the second experiment, the enthalpy of evaporation was determined measuring the heat drawn from a surface during evaporation. With a comparative measurement against methanol, the calculations for acetone, $\Delta_V H = 30.2 \pm 2.8 \text{ kJ/mol}$, and for n-hexane, $\Delta_V H = 32.0 \pm 1.9 \text{ kJ/mol}$, confirmed the previous results. To ensure the purity of the used chemicals, their specific density was verified via two different methods in addition to measurements of their respective refractive indexes.

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1 Introduction

In order to induce vaporization of a substance (the phase transition of liquid to gaseous *at* boiling temperature¹), heat must be provided to the system. Under the condition that vapor pressure p and temperature T remain constant during vaporization, the thermal energy provided to the system per mole of vaporized substance equals its molar enthalpy of vaporization $\Delta_V H$.

For the small temperature range of the experiments detailed further on, enthalpies of vaporization can be treated as material constants. Clausius-Clapeyron's model,

$$\frac{dp}{dT} = \frac{h^{(g)} - h^{(l)}}{T(v^{(g)} - v^{(l)})} \quad (1)$$

where $h^{(\varphi)}$ and $v^{(\varphi)}$ are the molar volumes of the substance in its liquid and gaseous phases φ , treats vapor pressure as a function of boiling temperature T with the coexistence curve between liquid and gaseous phases for a graph, and describes the change dp expected for a variation of dT in the substance's boiling temperature.

Taking into account that $v^{(g)} \gg v^{(l)}$, and provided the gaseous phase behaves according to ideal gas law

$$v^{(g)} = \frac{RT}{p} \quad (2)$$

the differential equation can be simplified to

$$\frac{dp}{p} = \frac{\Delta_V H}{R} \frac{dT}{T^2} \quad (3)$$

Integration yields

$$\ln\left(\frac{p}{p_0}\right) = \frac{\Delta_V H}{R} \left(\frac{1}{T_0} - \frac{1}{T}\right) \quad (4)$$

Where p_0 and T_0 are atmospheric pressure and normal boiling temperature at atmospheric pressure respectively and $\Delta_V H$

can be calculated with an arbitrary point (p, T) on the coexistence curve.

The substance's entropy of vaporization at normal boiling temperature can be calculated via the Gibbs-Helmholtz equation

$$\Delta G = \Delta_V H - T\Delta_V S \quad (5)$$

knowing that $\Delta G = 0$ for reactions that have reached an equilibrium.

A phase transitions from the liquid to the gaseous phase can also take place below boiling temperature and vapor pressure. This can be observed for example when blow-drying one's hair or upon stepping out of the water as the droplets on the skin gradually evaporate. This is possible, because energy levels of the molecules inside the substance differ and some molecules have enough energy to overcome the threshold of the intermolecular forces binding them to the liquid.

The molecule's transition from fluid to gaseous phase requires energy which is taken from the fluid in the form of thermal energy, causing a momentary drop in temperature. If the fluid shares an interface with another substance (for example the skin) which has a higher temperature, it will in turn draw energy from this surface to compensate for the deficit. Such changes in temperature are very minor for small volumes of evaporated liquid but can be measured for example if the interface is shared with a highly sensitive temperature sensor.

The recorded temperature drops at contact with the sample and return to its previous value once the entire liquid has evaporated, presenting as a downward peak in a diagram where the sensor's temperature is plotted against time. The area under the peak, established as A , which is calculated from the data obtained from the sensor, is proportional to the thermal energy taken by the liquid from the sensor during that time:

$$A = \int \Delta T dt \propto Q = n\Delta_V H \quad (6)$$

¹Not to confuse with evaporation, which is the phase transition *below* the boiling temperature of a substance.

where n is the amount of substance given in moles.

The molar enthalpy of evaporation of the substance can be calculated from this using a reference substance with a known molar enthalpy of evaporation as follows:

$$\Delta_V H_{\text{subst}} = \Delta_V H_{\text{ref}} \frac{\rho_{\text{ref}}}{\rho_{\text{subst}}} \frac{M_{\text{subst}}}{M_{\text{ref}}} \frac{A_{\text{subst}}}{A_{\text{ref}}} \quad (7)$$

Where ρ is the sample's density, determined separately or taken from known literature.

2 Experimental

2.1 Chemicals

The following experiments were conducted using acetone (58.8 g/mol), from Sigma Aldrich, puriss. p.a., ($\geq 99.5\%$), n-hexane (86.18 g/mol), from Sigma Aldrich, ($\geq 95\%$) and methanol (32.04 g/mol), from VWR Chemicals, ($\geq 99.5\%$). All chemicals were used forgoing any further purification.

2.2 Procedure

A total of 3 experiments were prepared and carried out as follows:

2.2.1 Verification

The identity and purity of acetone and n-hexane were verified by determining their respective *refractive index* and *density*.

Refractive index measurements were done via digital refractometer ATAGO RX-5000 operating at standard wavelength of 589.0 nm (D-line) and with samples maintained at a constant $20.00 \pm 0.02^\circ\text{C}$ by a LAUDA E100 circulation thermostat (attached to the refractometer). After ensuring the glass surface of the sample block was dry, a small amount of liquid was applied – just enough to cover the circular surface – and once equilibration was reached as indicated by the temperature display, measurements could be initiated.

Density was determined in two different ways.

First, by *method A*, filling a 25.00 ± 0.04 mL volumetric flask (insert mark) with either liquid and measuring its mass using a METTLER-TOLEDO AG204 Delta Range analytical balance, the accuracy range of which is stated as 0.1 mg by the manufacturer.

And secondly, by *method B*, using an ANTON PAAR DMA 48 density gauge (Fig. 10), where the respective sample was carefully inserted via plastic syringe and – after visual confirmation that there were no air bubbles inside the u-shaped glass tube – run at setting F505. Between measurements of different samples, the tube was rinsed with deionized water and dried by inducing airflow.

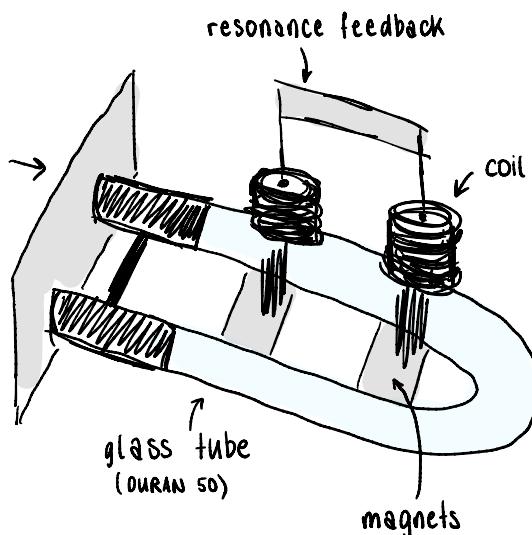


Figure 1: The ANTON PAAR DMA 48 density gauge measures a liquid's density by oscillating a glass tube. The resonant frequency of the glass tube is directly correlated to the liquid's density.

2.2.2 Vapor Pressure

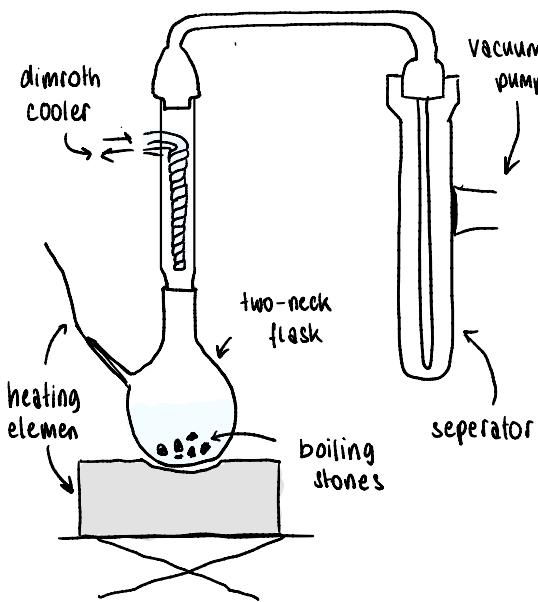


Figure 2: The vapor pressure measurement setup allows temperature and pressure to be controlled while observing the phase transition of the liquid.

The boiling temperatures of acetone and n-hexane were measured at different pressure settings.

For this purpose, a two necked flask containing the sample and about 10-15 boiling stones and filled to approximately half its capacity was attached to a pre-prepared setup (Fig.2), omitting grinding grease due to the risk of contaminating the sample and comparatively low significance of a tight seal in this experiment.

Only after ensuring that all openings were closed, the BÜCHI VAC V-503 vacuum pump, dimroth condensation cooler and WINKLER WHLG2 laboratory heating mantle with a WL10 heating controller were turned on. It is important to note that forgoing the former here and starting the cooler before sealing the system would allow water vapor from the air to condense inside the cooler and likely lead to falsified results.

While maintaining a low heat supply via the controllable heating mantle, pressure

within the system was steadily decreased under close surveillance through evacuation by means of a BÜCHI I-100 vacuum controller and a ventilation valve until about 150 mbar and 100 mbar were reached for acetone and n-hexane respectively and the liquid was simultaneously boiling inside the flask and dripping steadily from the cooler. For acetone we could not observe any dripping at first, yet the liquid seemed to be boiling and the temperature was quickly decreasing to under 10 °C. We concluded that the vapor currently forming would be at a lower temperature than the liquid in the dimroth cooler, causing it to escape into the separator instead of condensing. As such we increased our heat supply and not long after, equilibrium returned to the system and steady dripping could be observed.

The temperature measured by the GREISINGER GMH 3210 digital temperature gauge (with a resolution of 0.1 K) was marked down with its corresponding vapor pressure and the pressure was incremented in 25 mbar to 50 mbar steps until reaching 900 mbar, waiting for the temperature to level out after each increase and recording respective values. This was repeated at decreasing pressure, generating two sets of values for each sample.

2.2.3 Evaporation Cooling

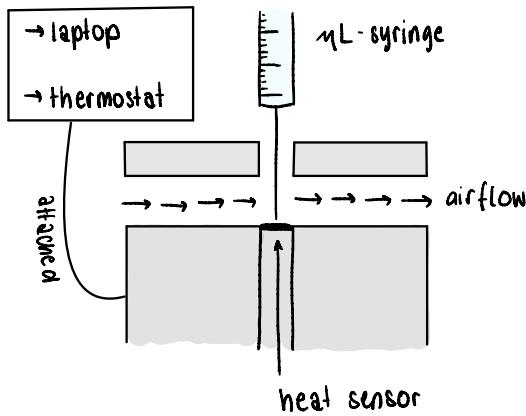


Figure 3: The TREVAC evaporation cooling measurement system causes the liquid to evaporate and draw heat from the sensor plate, resulting in a temporary temperature drop.

The evaporation cooling effect of acetone, n-hexane and methanol (reference sample) were visualized and recorded by measuring the surface temperature of an ultra-sensitive heat sensor at 0.25 s intervals during the evaporation of a predefined volume of liquid sample applied to the sensor.

The setup, a TREVAC-apparatus (transient evaporation cooling) as can be seen in Fig. 3, self-developed by the PCL at ETH Zürich, relies on a LAUDA Ecoline 103 thermostat to keep the aluminium block with the NATIONAL LM 35 heat sensor at its center at a programmable base temperature of

$T_0 = 35^\circ\text{C}$, deviating less than $\pm 0.01 \text{ K}$. T_0 was selected at 30 K to 40 K below the boiling point of the lowest-boiling liquid. The sample was inserted at a steady pace via a HAMILTON 801 RN microliter syringe (scale facing forward to ensure measurement circumstances were as similar as possible for each sample inserted), using a measuring gauge to measure out exactly 5 μL and discard any excess liquid beforehand.

The registered surface temperature over time was recorded via analog/digital con-

verter (ADC, 23 bit) and broadcast on the display of a laptop attached to the setup.

This process was repeated 3 times per substance, leaving enough time for the sensor to equilibrate back to T_0 between the end of each prior evaporation and insertion of the current sample.

3 Results and Discussion

The analysis and calculations were conducted using the R programming language [5] and Python Jupyter Notebooks [4]. The scripts are included in the appendix of this report. A.1 All uncertainties stated are provided for a 95 % confidence interval.

3.1 Density and refractive index

The refractive index and density of acetone were measured at

$$n_D^{20} = 1.3591 \text{ and}$$

$$\rho = 0.791 \text{ g/mL (method B)}$$

and the density of acetone was calculated (method A) to be

$$0.7904 \pm 0.0026 \text{ g/mL}$$

corresponding with literature values of

$$n_D^{20} = 1.3591 \text{ and}$$

$$\rho = 0.790 \text{ g/mL. [1]}$$

For n-hexane values of

$$n_D^{20} = 1.3751 \text{ and}$$

$$\rho = 0.659 \text{ g/mL (method B) and}$$

$$\rho = 0.6572 \pm 0.0022 \text{ g/mL (method A)}$$

were obtained compared to literature values of

$$n_D^{20} = 1.3751 \text{ and}$$

$$\rho = 0.660 \text{ g/mL. [1]}$$

It can be observed, that the results obtained for acetone are more precise than those of n-hexane. This could be explained for example by the varying grades of purity (99.5% vs. 95% specified by their producers).

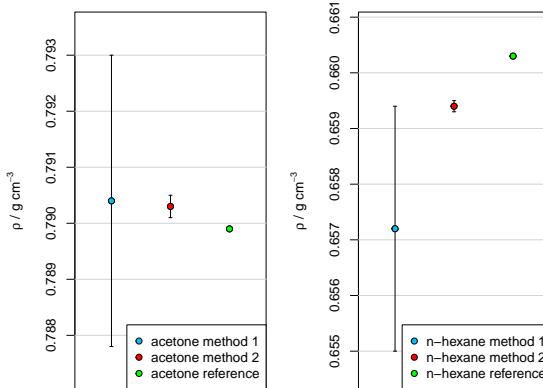


Figure 4: Comparisons of the density values measured, and reference values found in literature [1].

3.2 Vapor Pressure

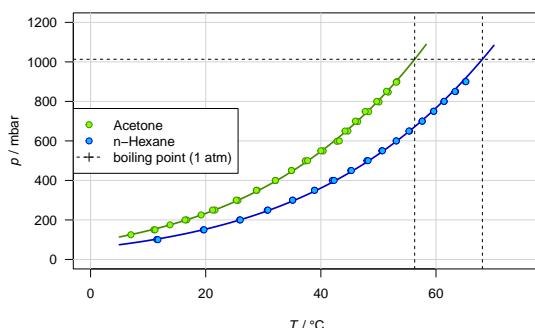


Figure 5: The vapor pressure curves illustrate the exponential correlation between vapor pressure and temperature. (Standard boiling points extrapolated and marked by the vertical and horizontal lines.)

As mentioned in the introduction, vapor pressure can be described as $p(T)$. Accordingly, the results obtained from measuring the samples' boiling temperatures at differing surrounding pressures were plotted in a p - T -diagram, presented in Fig. 5.

Enthalpies of vaporization were calculated with by plotting logarithmic $\frac{p}{p_0}$ against inverse temperature T via a linear regression model (Fig. 6) and calculating the slope b of the resulting linear graphs. Expansion of

equation (1) shows that it is equal to $\frac{\Delta_V H}{R}$. Results amounted to

$$\Delta_V H = 32.49 \pm 0.26 \text{ kJ/mol for acetone}$$

$$\Delta_V H = 32.66 \pm 0.25 \text{ kJ/mol for n-hexane.}$$

Acetone is a polar solvent and n-hexane is not. Because of this, one would assume, that its enthalpy of vaporization and standard boiling temperature would be higher than those of n-hexane because of stronger intermolecular bonds. Evidently, this is not the case. A possible explanation could be that though it is polar, acetone has a lower molar mass than n-hexane, meaning it would take less energy to bring one mol of acetone from its fluid to its gaseous form than one mol of another substance with similar polarity.

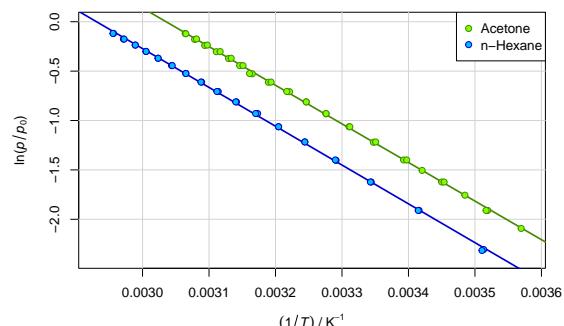


Figure 6: The Enthalpy of vaporization for each sample can be visualized in a $\ln\left(\frac{p}{p_0}\right) - \frac{1}{T}$ diagram, because it is directly proportionate to the linear graph's slope b (by factor of the gas constant R .)

Normal boiling temperatures T_0 and standard entropies $\Delta_V S(T_0)$ of vaporization were also calculated with the help of the linear model parameters intercept a and slope b and found to be

$$T_0^{\text{acet}} = 56.29 \text{ C}$$

$$T_0^{\text{nhex}} = 68.06 \text{ C}$$

corresponding to literature values of

$$T_0^{\text{acet}} = 56.15 \pm 0.30 \text{ C}$$

$$T_0^{\text{nhex}} = 68.75 \pm 0.30 \text{ C}$$

and

$$\Delta_V S(T_0)^{\text{acet}} = 98.62 \text{ J}/(\text{mol K})$$
$$\Delta_V S(T_0)^{\text{nhex}} = 95.74 \text{ J}/(\text{mol K}).$$

3.3 Transient evaporation cooling

The data files generated by the TREVAC apparatus were imported to R. With the `identify()` command, the relevant time ranges for the peak integration could be selected graphically. (Fig. 7) This data, including statistical parameters were then exported to a *csv* file for documentation purposes and for further processing. To calculate the final values, including standard errors, a Jupyter notebook [4] with the METAS UncLib uncertainty modeling software [6] was used. Formula 7 was

For acetone, $30.2 \pm 2.8 \text{ kJ/mol}$ was calculated, and for n-hexane, $32.0 \pm 1.9 \text{ kJ/mol}$, was found.

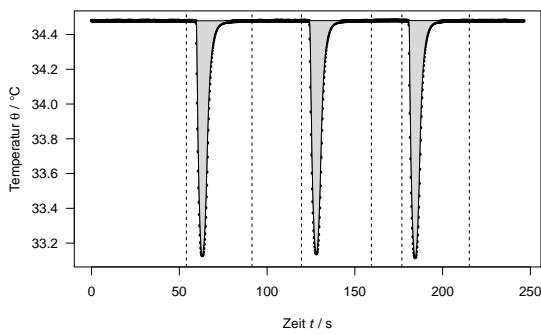


Figure 7: Temperature peaks caused by n-hexane withdrawing heat from the measurement surface. The grey area is directly correlating with the enthalpy of evaporation $\Delta_V H_{\text{subst}}$ of the substance.

Standard values for $\Delta_V H$ can be found on the website of the National Institute of Standard Technology:

$$\Delta_V H_{\text{acet}}^0 = 31.27 \text{ kJ/mol} [2]$$
$$\Delta_V H_{\text{nhex}}^0 = 31 \pm 1 \text{ kJ/mol} [3]$$

References

- [1] Erich Meister. *Praktische Allgemeine Chemie*. 2023.
- [2] NIST Chemistry WebBook/Acetone. URL: <https://webbook.nist.gov/cgi/cbook.cgi?ID=C67641&Mask=4#Thermo-Phase>. (accessed: 12.01.2024).
- [3] NIST Chemistry WebBook/n-Hexane. URL: <https://webbook.nist.gov/cgi/cbook.cgi?ID=C110543&Mask=4#Thermo-Phase>. (accessed: 12.01.2024).
- [4] Fernando Pérez and Brian E. Granger. “IPython: a System for Interactive Scientific Computing”. In: *Computing in Science and Engineering* 9.3 (May 2007), pp. 21–29. ISSN: 1521-9615. DOI: 10.1109/MCSE.2007.53. URL: <https://ipython.org/>.
- [5] R Core Team. *R: A Language and Environment for Statistical Computing*. R Foundation for Statistical Computing. Vienna, Austria, 2021. URL: <https://www.R-project.org/>.
- [6] Michael Wollensack. *METAS UncLib*. URL: <https://www.metas.ch/metas/en/home/fabe/hochfrequenz/unclib.html>. (accessed: 12.01.2024).

A Appendix

A.1 R-Scripts

A.1.1 Vapor Pressure Analysis

```
rm(list=ls())

library(readxl)

R <- 8.314462618      # J K^-1 mol^-1
p0 <- 1013.25

COL_ACET_PRIM <- "#458B00"
COL_ACET_SEC <- "#7FFF00"

COL_NHEX_PRIM <- "#0000CD"
COL_NHEX_SEC <- "deepskyblue"

WIDTH <- 7
HEIGHT <- 5

quartz(height=HEIGHT, width=WIDTH)

# hack copied from https://stackoverflow.com/questions/1826519/how-to-
# assign-from-a-function-which-returns-more-than-one-value
':=' <- function(lhs, rhs) {
  frame <- parent.frame()
  lhs <- as.list(substitute(lhs))
  if (length(lhs) > 1)
    lhs <- lhs[-1]
  if (length(lhs) == 1) {
    do.call(`=`, list(lhs[[1]], rhs), envir=frame)
    return(invisible(NULL))
  }
  if (is.function(rhs) || is(rhs, 'formula'))
    rhs <- list(rhs)
  if (length(lhs) > length(rhs))
    rhs <- c(rhs, rep(list(NULL), length(lhs) - length(rhs)))
  for (i in 1:length(lhs))
    do.call(`=`, list(lhs[[i]], rhs[[i]]), envir=frame)
  return(invisible(NULL))
}

functionReturningTwoValues <- function() {
  return(list(1, matrix(0, 2, 2)))
}
c(a, b) := functionReturningTwoValues()
```

```
digest_file <- function(filename, sheet) {  
  
  raw_data = read_xlsx(path=filename, sheet=sheet)  
  
  # for plot invt-lnp  
  p = unlist(raw_data["p/mbar"])  
  t = unlist(raw_data["T/°C"])  
  lnp = log(p/p0)  
  tk = t + 273.15    # t in K  
  rez_tk = 1/tk  
  
  sp1 = p  
  sp2 = round(t, digits=1)  
  sp3 = signif(rez_tk, digits=4)  
  sp4 = round(lnp, digits=4)  
  
  table_1 = data.frame(sp1,sp2,sp3,sp4, row.names=NULL)  
  names(table_1) = c("p/mbar", "T/°C", "(1/T)/K^-1", "ln(p/p0)")  
  print(table_1)  
  
  gerade = lm(lnp ~ rez_tk)  
  print(summary(gerade))  
  a = summary(gerade)$coef[1,1]  
  b = summary(gerade)$coef[2,1]  
  sa = summary(gerade)$coef[1,2] # Standardfehler davon  
  sb = summary(gerade)$coef[2,2]  
  
  # for plot t-p  
  dof = length(unlist(raw_data["p/mbar"]))-2 # degrees of freedom  
  ts = qt(0.975, dof)    # ts Fraktile  
  d_vH = -R * b          # molare Verdampfungsenthalpie  
  c_d_vH = R * sb * ts   # entsprechendes Vertrauensintervall  
  
  ntk = -b/a            # Normalsiedetemp. in K  
  d_vS = R * a           # molare Verdampfungsentropie bei ntk  
  
  table_2 = data.frame(d_vH, c_d_vH, ntk, d_vS)  
  names(table_2) = c("d_vH", "c_d_vH", "ntk", "d_vS")  
  print(table_2)  
  
  t_curve = seq(from=5,to=ntk-273.15+2, length=201)  
  p_curve = p0*exp(a + b/(t_curve + 273.15))  
  
  return(list(  
}
```

```
rez_tk,  
lnp,  
gerade,  
  
t,  
p,  
ntk,  
t_curve,  
p_curve  
))  
  
}  
  
ddr <- function(filename, sheet_acet, sheet_nhex, export_name_inv_ln,  
← export_name_tp){  
  
# -- digest excel files --  
# acetone  
c(  
  acet_rez_tk,  
  acet_lnp,  
  acet_gerade,  
  
  acet_t,  
  acet_p,  
  acet_ntk,  
  acet_t_curve,  
  acet_p_curve  
) := digest_file(filename, sheet_acet)  
  
# n-hexane  
c(  
  nhex_rez_tk,  
  nhex_lnp,  
  nhex_gerade,  
  
  nhex_t,  
  nhex_p,  
  nhex_ntk,  
  nhex_t_curve,  
  nhex_p_curve  
) := digest_file(filename, sheet_nhex)  
  
# plot rez_tk / lnp  
plot(acet_rez_tk, acet_lnp, type="n",  
      xlim=c(0.00293, 0.00358),  
      ylim=c(-2.4, 0.0),
```

```
xlab=expression((1/italic(T))*" / "*K^-1),
ylab=expression("ln(*italic(p/p[0])*")")
)

grid(nx = NULL, ny = NULL,
      lty = 1,          # Grid line type
      col = "lightgray", # Grid line color
      lwd = 1)          # Grid line width

abline(acet_gerade, lwd=2, col=COL_ACET_PRIM)
points(acet_rez_tk, acet_lnp, type="p", pch=21, bg=COL_ACET_SEC,
       col=COL_ACET_PRIM)

abline(nhex_gerade, lwd=2, col=COL_NHEX_PRIM)
points(nhex_rez_tk, nhex_lnp, type="p", pch=21, bg=COL_NHEX_SEC,
       col=COL_NHEX_PRIM)

legend(
  "topright",
  legend=c(
    "Acetone", "n-Hexane"
  ),
  col=c(COL_ACET_PRIM, COL_NHEX_PRIM),
  pt.bg=c(COL_ACET_SEC, COL_NHEX_SEC),
  pch=c( 21,21),
  bg="white"
)

dev.copy2pdf(file=export_name_inv_ln, width=WIDTH)

plot(acet_t, acet_p, type="n", las=1,
      xlim=c(0,nhex_ntk-273.15+7),
      ylim=c(0,1200),
      xlab=expression(italic(T)*" / "*`^oC`),
      ylab=expression(italic(p)*" / "*`mbar`)
)
grid(nx = NULL, ny = NULL,
      lty = 1,          # Grid line type
      col = "lightgray", # Grid line color
      lwd = 1)          # Grid line width

# acetone
lines(acet_t_curve, acet_p_curve, lwd=2, col=COL_ACET_PRIM)
points(acet_t, acet_p, type="p", pch = 21, bg=COL_ACET_SEC,
       col=COL_ACET_PRIM)
abline(v=acet_ntk-273.15, lty=2)
abline(h=p0, lty=2)
```

```
lines(nhex_t_curve, nhex_p_curve, lwd=2, col=COL_NHEX_PRIM)
points(nhex_t, nhex_p, type="p", pch = 21, bg=COL_NHEX_SEC,
       col=COL_NHEX_PRIM)
abline(v=nhex_ntk-273.15, lty=2)

legend(
  "left",
  legend=c(
    "Acetone", "n-Hexane", "boiling point (1 atm)"
  ),
  col=c(COL_ACET_PRIM, COL_NHEX_PRIM, "black"),
  pt.bg=c(COL_ACET_SEC, COL_NHEX_SEC),
  pch=c(21,21, 3),
  lty=c(0,0,2),
  bg="white"
)

dev.copy2pdf(file=export_name_tp, width=WIDTH)
}

#ddr("DDR_Messdaten_1.xlsx", "n-Hexane (all)", "Fig_DDR1_nhex_1.pdf",
#      "Fig_DDR1_nhex_2.pdf")

ddr(
  filename="DDR_Messdaten_1.xlsx",
  sheet_acet="Acetone (all)",
  sheet_nhex="n-Hexane (all)",
  export_name_inv_ln="ddr1_figs/DDR1_inv_ln.pdf",
  export_name_tp="ddr1_figs/DDR1_t_p.pdf"
)
```

A.1.2 TREVAC Analysis

```
rm(list=ls())

library("MASS")

NPEAKS = 3
SAVE <- FALSE

PeakIntegral <- function(ind, Zeit, Temp) {

  # select index in time range
  # idx <- which(Zeit>Zeitbereich[1] & Zeit<Zeitbereich[2])

  # subset time/theta
  t <- Zeit[ind[1]:ind[2]]
  theta <- Temp[ind[1]:ind[2]]

  theta0 <- theta[1]
  A <- 0

  for (i in 1:(length(t) - 1)) {
    A <- A +
      (theta[i] + theta[i + 1]) / 2 - theta0
    ) * (t[i+1] - t[i])
  }

  abline(v=range(t), lty=2)
  polygon(c(t, rev(t)), c(theta, 0*theta+theta0), col="grey85")
  A
}

trans_enthalp <- function (filename, save_to) {

  Messdaten <- read.table(paste("DDR_Messdaten_2/", filename, sep=""))

  Zeit <- Messdaten[,1]
  Temp <- Messdaten[,2]

  plot(
    Zeit, Temp,
    type="o", pch=16, cex=0.6, las=1,
    xlab=expression("Zeit " * italic(t) * " / s"),
    ylab=expression("Temperatur " * theta * " / " * degree * "C")
  )

  indexes = matrix(integer(), nrow=2, ncol=0)
```

```
Areas = c()

for (i in 1:NPEAKS) {
  ind = sort(identify(Zeit, Temp, n=2, plot=FALSE))
  indexes = cbind(indexes, ind)
  print(indexes)

  Areas = cbind(Areas, PeakIntegral(ind, Zeit, Temp))

}

if(SAVE) {
  dev.copy2pdf(file=paste(save_to, ".pdf", sep=""), width=7, height=5)
  write.matrix(indexes, file=paste(save_to, "_indexes.csv", sep=""))
  write.table(Areas, file=paste(save_to, "_areas.csv", sep=""))
}

trans_enthalp("methanolmzjj.dat", "ddr2_exports/methanol")
trans_enthalp("acetone-2-mzjj.dat", "ddr2_exports/acetone")
#trans_enthalp("acetone-mzjj.dat", "ddr2_exports/acetone-old")
trans_enthalp("n-hexane-mzjj.dat", "ddr2_exports/n-hexane")
```

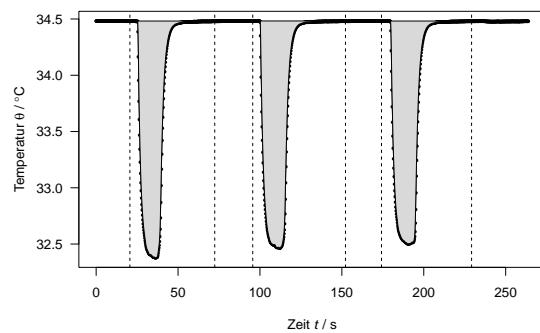


Figure 8: TREVAC area analysis of methanol as reference value.

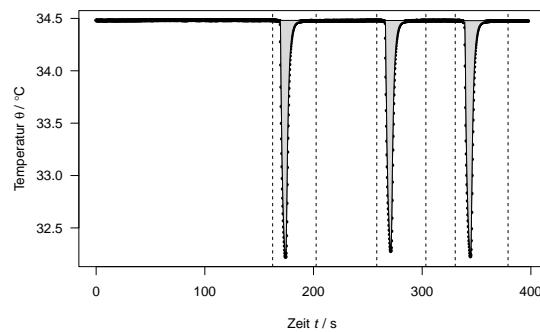


Figure 9: TREVAC area analysis of acetone.

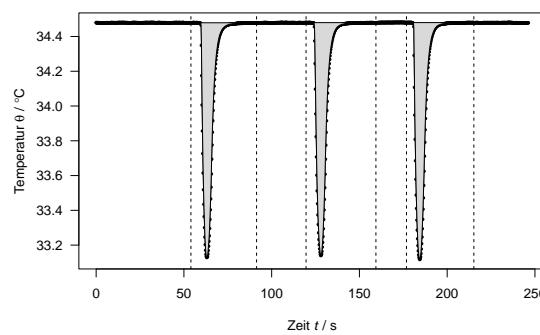


Figure 10: TREVAC area analysis of n-hexane.

A.1.3 TREVAC results and uncertainty estimates

```
In [1]: from IPython.display import display, Math
In [2]: import math
In [3]: import pandas as pd
/Users/Janjo/opt/anaconda3/lib/python3.8/site-packages/pandas/core/computation/expressions.py:20: UserWarning: Pandas requires version '2.7.3' or newer of 'numexpr' (version '2.7.1' currently installed).
         from pandas.core.computation.check import NUMEXPR_INSTALLED
In [4]: # METAS UncLib
        from metas_unclib import * # import METAS UncLib
        use_linprop() # linear unc propagation
In [5]: from Metas.UncLib.LinProp import UncBudget as _LinPropUncBudget
In [6]: # unc_budget bug fix

def unc_budget(unc_item):
    tree = _LinPropUncBudget.ComputeTreeUncBudget(unc_item.net_object)

    data = np.zeros((len(tree) + 1, 2))
    desc = [""]*(len(tree) + 1)

    desc[0] = "SUMMARY"
    data[0] = (
        unc_item.stdunc,
        100.,
    )

    for i, elem in enumerate(tree):
        data[i + 1] = (
            elem.get_UncComponent(),
            elem.get_UncPercentage(),
        )
        desc[i + 1] = elem.get_Description()

    try:
        # render pandas.DataFrame
        import pandas as pd
        return pd.DataFrame(columns=("description", "unc component", "unc / %"), data={
            "description": desc,
            "unc component": data[:,0],
            "unc / %": data[:,1],
        }).sort_values("unc / %", ascending=False)
    except ModuleNotFoundError:
        print("unc component | unc / % | label")
        print("-----|-----|-----")
        for label, (component, percent) in zip(desc, data):
            print("{:.4E} | {:.4E} | {}".format(component, percent, label))
    return data, desc
```

Formelübersicht

$$\Delta_V H_{\text{subst}} = \Delta_V H_{\text{ref}} \frac{\rho_{\text{ref}}}{\rho_{\text{subst}}} \frac{M_{\text{subst}}}{M_{\text{ref}}} \frac{A_{\text{subst}}}{A_{\text{ref}}}$$

$$\int \Delta T dt = A \propto Q = n \Delta_V H$$

Q : Verdunstungswärme

n : Stoffmenge

M : Molmasse

ρ : spezifische Dichte

$\Delta_V H$: molare Verdampfungsenthalpie

A : Peak-Fläche

Funktionen

```
In [7]: def calc_h(H_ref, rho_ref, rho_subst, M_ref, M_subst, A_ref, A_subst):
    return (
        H_ref * (rho_ref / rho_subst)
        * (M_subst / M_ref)
        * (A_subst / A_ref)
    )
```

```
In [8]: def get_areas(path):
    return list(pd.read_csv(path, sep=" ")).iloc[0])
```

Measured values and constants

```
In [9]: # rho values literature [kg/dm3]
rho_lit_m = ufloat(0.7914, desc="rho methanol (literature)")
rho_lit_a = ufloat(0.7899, desc="rho acetone (literature)")
rho_lit_n = ufloat(0.6603, desc="rho n-hexane (n-hexane)")
```

```
In [10]: # uncertainties of devices
u_m = 0.002 # [g]
u_V = 0.08 # [mL]
```

```
In [11]: # density acetone measured in exercise 3
m_a = ufloat(19.76, u_m, desc="mass acetone") # [g]
V_a = ufloat(25., u_V, desc="volume acetone") # [mL]
rho_a = m_a / V_a # [kg/dm3]
rho_a
```

```
Out[11]: 0.7904000000000001 ± 0.0025305448659132686
```

```
In [12]: # acetone calculated with method of the Meister book
1/V_a.value * u_m + abs(-m_a.value / V_a.value**2) * u_V # [kg/dm3]
```

```
Out[12]: 0.0026092800000000007
```

```
In [13]: # density n-hexane measured in exercise 3
m_n = ufloat(16.45, u_m, desc="mass n-hexane") # [g]
V_n = ufloat(25., u_V, desc="volume n-hexane") # [mL]
rho_n = m_n / V_n # [kg/dm3]
rho_n
```

```
Out[13]: 0.6579999999999999 ± 0.002107119208777709
```

```
In [14]: # n-hexane calculated with method of the Meister book
1/V_n.value * u_m + abs(-m_n.value / V_n.value**2) * u_V # [kg/dm3]
```

```
Out[14]: 0.0021856
```

```
In [15]: # Molar masses [kg/mol]
M_m = ufloat(32.04, desc="M methanol")
M_a = ufloat(58.08, desc="M acetone")
M_n = ufloat(86.18, desc="M acetone")

In [16]: # evaporation enthalpy
H_m = ufloat(37.43, 0.17, desc="H methanol")
H_m

Out[16]: 37.43 ± 0.17
```

Reference: Methanol

```
In [17]: # read in measured areas
met_samples = get_areas("../ddr2_exports/methanol_areas.csv")
met_samples

Out[17]: [-28.655065, -29.9144020000002, -29.660473]

In [18]: # transform to uncertainty object
A_met = ufloatfromsamples(met_samples, desc="are methanol")
A_met

Out[18]: -29.409980000000065 ± 0.8441024984395747
```

Acetone

```
In [19]: # read in measured areas
ac_samples = get_areas("../ddr2_exports/acetone_areas.csv")
ac_samples

Out[19]: [-12.963891, -12.7988540000001, -13.5183319999999]

In [20]: # transform to uncertainty object
A_ac = ufloatfromsamples(ac_samples, desc="area acetone")
A_ac

Out[20]: -13.093692333333335 ± 0.4776896236957493

In [21]: # calculate evaporation enthalpy of acetone
H_ac = calc_h(
    H_ref=H_m,
    rho_ref=rho_lit_m,
    rho_subst=rho_a,
    #rho_subst=rho_lit_a,
    M_subst=M_a,
    M_ref=M_m,
    A_ref=A_met,
    A_subst=A_ac,
) # [kJ/mol]
H_ac

Out[21]: 30.24617388090784 ± 1.4140226667388984

In [22]: H_ac.stdunc

Out[22]: 1.4140226667388984

In [23]: H_ac_result = r"\Delta VH_{\text{acetone}} = {:.1f} \pm {:.1f} \text{ kJ/mol } (95\%)"
display(Math(H_ac_result))
```

$$\Delta VH_{\text{acetone}} = 30.2 \pm 2.8 \text{ kJ/mol (95 \%)}$$

```
In [24]: print(H_ac_result)
\Delta_VH_{\text{acetone}} = 30.2 \pm 2.8 \text{ kJ/mol } (95 \text{ space \%})
```

n-Hexane

```
In [25]: # read in measured areas
n_hex_samples = get_areas("../ddr2_exports/n-hexane_areas.csv")
n_hex_samples
```

```
Out[25]: [-7.736397999999, -7.7719409999995, -7.7942649999992]
```

```
In [26]: # transform to uncertainty object
A_nhex = ufloatfromsamples(n_hex_samples, desc="area n-hexane")
A_nhex
```

```
Out[26]: -7.767534666666591 ± 0.036989056992450586
```

```
In [27]: # calculate evaporation enthalpy of n-hexane
H_nhex = calc_h(
    H_ref=H_m,
    rho_ref=rho_lit_m,
    rho_subst=rho_n,
    #rho_subst=rho_lit_n,
    M_subst=M_n,
    M_ref=M_m,
    A_ref=A_met,
    A_subst=A_nhex,
) # [kJ/mol]
H_nhex
```

```
Out[27]: 31.981028970326822 ± 0.9472648790759943
```

```
In [28]: H_nhex_result = r"\Delta_VH_{\text{n-hexane}} = {:.1f} \pm {:.1f} \text{ kJ/mol }"
display(Math(H_nhex_result))
```

$$\Delta_VH_{\text{n-hexane}} = 32.0 \pm 1.9 \text{ kJ/mol (95 \%)}$$

```
In [29]: print(H_nhex_result)
\Delta_VH_{\text{n-hexane}} = 32.0 \pm 1.9 \text{ kJ/mol (95 \space \%)} 
```

```
In [30]: unc_budget(H_nhex)
```

	description	unc component	unc / %
0	SUMMARY	0.947265	100.000000
1	are methanol	0.917895	93.895095
2	area n-hexane	0.152294	2.584771
3	H methanol	0.145252	2.351257
5	volume n-hexane	0.102339	1.167191
4	mass n-hexane	0.003888	0.001685

A.1.4 Density comparison plot

```
rm(list = ls()) # tabula rasa

source("../KAL_Auswertung/helpers.R")

HEIGHT <- 5
WIDTH <- 7


par(
  mfrow=c(1,2),
  mar=c(0.7, 5,1,0.7)
)

acet.rho.data = c(
  0.7904, # acetone measured method 1
  0.7903, # acetone measured method 2
  0.7899 # acetone source meister
)

nhex.rho.data = c(
  0.6572, # n-hex measured method 1
  0.6594, # n-hex measured method 2
  0.6603 # n-hex source meister
)

acet.rho.se = c(
  0.0026, # acetone measured method 1
  0.0002, # acetone measured method 2
  1e-5 # acetone source meister
)

nhex.rho.se = c(
  0.0022, # n-hex measured method 1
  0.0001, # n-hex measured method 2
  1e-5 # n-hex source meister
)

plot.colrcycle = c("deepskyblue", "red", "green")

plot(
  acet.rho.data,
  xlab="",
  ylab=expression(italic(rho)*" / g "*cm^-3),
  xaxt='n',
  xlim=c(0.5, 3.5),
```

```
ylim=c(acet.rho.data[1] - 1.2 * acet.rho.se[1], acet.rho.data[1] + 1.2 *
       ↵ acet.rho.se[1])
)

plot.grid(nx=NA)

FBy(
  1:3,
  acet.rho.data,
  acet.rho.se,
  bg=plot.colortable
)

legend(
  "bottomright",
  legend=c(
    "acetone method 1", "acetone method 2", "acetone reference"
  ),
  #col=c(COL_ACET_PRIM, COL_NHEX_PRIM, "black"),
  pt.bg=plot.colortable,
  pch=c( 21,21, 21),
  lty=c(0,0,0),
  bg="white"
)
)

plot(
  nhex.rho.data,
  xlab="",
  ylab=expression(italic(rho)*" / g "*cm^-3),
  xaxt='n',
  xlim=c(0.5, 3.5),
  ylim=c(nhex.rho.data[1] - 1.2 * nhex.rho.se[1], nhex.rho.data[1] + 1.2 *
         ↵ nhex.rho.se[1] + 0.001)
)
)

plot.grid(nx=NA)

FBy(
  1:3,
  nhex.rho.data,
  nhex.rho.se,
  bg=plot.colortable
)

legend(
  "bottomright",
```

```
legend=c(  
  "n-hexane method 1", "n-hexane method 2", "n-hexane reference"  
)  
#col=c(COL_ACET_PRIM, COL_NHEX_PRIM, "black"),  
pt.bg=plot.colrcycle,  
pch=c( 21,21, 21),  
lty=c(0,0,0),  
bg="white"  
)  
  
plot.save("../Reports/", "rho-comparison.pdf")
```

A.1.5 Plotting helper scripts

```
# by github.com/janjoch, 2023

library(latex2exp)

# HELPER FUNCTIONS
join <- function(...) {
  paste(..., sep="")
}

# hack copied from https://stackoverflow.com/questions/1826519/how-to-
# assign-from-a-function-which-returns-more-than-one-value
':=' <- function(lhs, rhs) {
  frame <- parent.frame()
  lhs <- as.list(substitute(lhs))
  if (length(lhs) > 1)
    lhs <- lhs[-1]
  if (length(lhs) == 1) {
    do.call(`=`, list(lhs[[1]], rhs), envir=frame)
    return(invisible(NULL))
  }
  if (is.function(rhs) || is(rhs, 'formula'))
    rhs <- list(rhs)
  if (length(lhs) > length(rhs))
    rhs <- c(rhs, rep(list(NULL), length(lhs) - length(rhs)))
  for (i in 1:length(lhs))
    do.call(`=`, list(lhs[[i]], rhs[[i]]), envir=frame)
  return(invisible(NULL))
}

# usage
# func_that_returns_three_values <- function(a,b,c) {
#   return(list(a,b,c))
# }
# c(a,b,c) := func_that_returns_three_values(1,2,3)

FBy <- function(x, y, sy, ...) {
  # copied from Meister
  arrows(x, y - sy, x, y + sy, code=3, angle=90, length=0.02)
  points(x, y, pch=21, ...)
}

# PLOT MODULAR FUNCTIONS
plot.init.grey <- function(x, y) {
```

```
plot(x, y,
      type = "l",
      lwd=2,
      col="darkgrey",
      xlab=expression(italic(t)*" / "*s),
      ylab=expression(italic(T)*" / "*C))
)
plot.grid()
}

plot.grid <- function(nx=NULL, ny=NULL, lty=1, col="lightgray", lwd=1,
← ... ) {
  grid(
    nx = nx,
    ny = ny,
    lty = lty,      # Grid line type
    col = col, # Grid line color
    lwd = lwd,      # Grid line width
    ...
  )
}

plot.line.highlight <- function(x, y, col="blue") {
  lines(
    x,
    y,
    col=col,
    lw=3
  )
}

plot.line.annot <- function(x, y) {
  lines(
    x,
    y,
    lty=3,
    lw=2,
    col="black"
  )
}

plot.annot <- function(x, y, text, xjust=0.5, yjust=0.5, ...) {
  legend(
    x,
```

```
y,  
text,  
bg="white",  
box.col="white",  
adj=0.15,  
xjust=xjust,  
yjust=yjust,  
...  
)  
}  
  
plot.save <- function(export.path, export.plot) {  
  dev.copy2pdf(file=join(export.path, export.plot), width=WIDTH,  
  ↳ height=HEIGHT)  
}
```

A.2 Lab Journals

BOOK PAGE 1

TITLE DDR von reinen Flüssigkeiten PROJECT PC Praktikum 1. Semeskr DATE Jan. 3. 2024

(Continued from Page) Praktikumsbuch Kapitel 6, S. 73 ff. (Versuch 3) 2a. Auflage 2023

CHARAKTERISIERUNG VON FLÜSSIGKEITEN DURCH DICHTE & BRECHUNGSINDEX

* Ziel: Messung von Dichte & und Brechungsindex versch. Flüssigkeiten zur Verifizierung von Identität und Reinheit.

① Dichte: → Die Dichte wird bestimmt, indem nach $\rho = \frac{m}{V}$ Masse und Volumen der Flüssigkeit gemessen werden. die Masse der Flüssigkeit bei festgelegtem Volumen gemessen wird.
 ↗ Biegeschwinger-
 → Die Dichte wird durch ein Dichtemessgerät bestimmt 2a. Auflage 2023

* zu erwartende Dichte ρ [g/cm³] bei 20°C (aus Tabelle: Praktikumsbuch S. 10)
 - Aceton: 0,7899 g/cm³ - n-Hexan: 0,6603 g/cm³

② Brechungsindex: → Messung durch Digital-Refraktometer bei 20°C & 589,0 nm Wellenlänge
 * zu erwartende Brechungsindizes n_D^{20} bei 20°C & 589,0 nm λ (OMS: siehe Dichte oben)
 - Aceton: 1,3588 - n-Hexan: 1,3751

* Messgeräte:
 - Analysenwaage (METTLER-TOLEDO AG204 Delta Range), Unsicherheit: $\pm 1\text{mg}$
 - 25 mL Messkolben Unsicherheit: $\pm 0,04\text{ mL}$ (Praktikumsbuch S. 68)
 - Biegeschwinger-Dichtemessgerät (ANTON PAAR DMA 48)
 - Digital-Refraktometer (ATAGO RX-5000) oder nicht?

* Skizze Dichtemessgerät:

Continued to Page

SIGNATURE	DATE	WITNESS	DATE
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BOOK	PAGE	TITLE	PROJECT	DATE
		Continued from Page		
		* Skizze Refraktometer.		
15		* Chemikalien:	<ul style="list-style-type: none"> Aceton $(\text{CH}_3)_2 \text{CO}$ M = 58,08 n-Hexan C_6H_{14} M = 86,18 	<ul style="list-style-type: none"> → Tabelle S. 10 → Richtlinienbuch
16			<ul style="list-style-type: none"> ↳ CAS : 67-64-1 ↳ Sigma-Aldrich, puiss. 99,5%. 	
17			<ul style="list-style-type: none"> ↳ H 225 : Flüssig & gasförmig, hochentflammbar (2) geringe Anfälligkeit (2) ↳ H 319 : Schwere Augenschädigung (2) ↳ H 336 : Spezif. Zielorgan-Toxizität (3) einmalige Exposition (3) (narkotisierende Wirkung, Sonnenblindheit) 	<ul style="list-style-type: none"> ↳ H 225 : siehe oben (2) ↳ H 319 : Ätz-/Reizwirkung auf die Haut (2) ↳ H 361f: Reproduktionstoxizität (kann vermutl. Fruchtbarkeit beeinträchtigen (2)) ↳ H 336 : (3)
18		Gefahrenhinweise	<ul style="list-style-type: none"> ↳ Piktogramme: GHS02, GHS07 	<ul style="list-style-type: none"> ↳ H 373 : Kann Organe schädigen (Nervensystem (bei wiederholter Exposition)) ↳ H 304 : Aspirationsgefahr (bei Verschlucken o. Einatmen sehr tödlich) ↳ H 411 : Gewässergefährdet
20				
21		Sicherheitshinweise	<ul style="list-style-type: none"> ↳ P 210 : Von Hitze, offener Flamme, heißen Oberflächen fernhalten, (nicht Rauchen) ↳ P 305 : Bei Kontakt mit den Augen einige Minuten lang mit H_2O spülen (wte. Linsen nach Möglichkeit entfernen) weiterspülen 	<ul style="list-style-type: none"> ↳ Piktogramme: GHS02, GHS07, OSE, O9 ↳ P 202 : Sicherheitshinw. vor Gebrauch lesen ↳ P 280 : Schutzhandschuhe / Brillen tragen ↳ P 303 : Bei Berührung mit Haut od. Haar 361 alle Kleidungsstücke ausschütteln & 351 Haut mit H_2O abwaschen ↳ P 304 : Bei Einatmen: sicheren, frische Luft, ungenießbare Atmung ↳ P 308 : Bei Exposition: ärztliche Hilfe konsultieren
22				
23		Lagerung/Safety	<ul style="list-style-type: none"> { P 403 } An gut belüfteten Ort aufbewahren (nicht verschlossener Behälter) 	<ul style="list-style-type: none"> → nach Verw. Abspülung mit Aceton.
24				
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		Continued to Page		
SIGNATURE	DATE	WITNESS	DATE	

TITLE		PROJECT		BOOK PAGE								
Continued from Page				DATE								
<p>Vorgenommen: ① (sauberer & trockener) 25 mL Messkolben auf Analysenwaage stellen $\Rightarrow \frac{1}{V} \cdot u_m + \frac{m}{V^2} u_v$ TARE drücken, Messkolben mit jeweiliger Flüssigkeit füllen, wieder auf Waage, Masse ablesen mit $u_m = 0,002$ $(2 \times 1 \text{ mg}) + \text{un-gen} \pm 8$ $\hookrightarrow u_v = 0,008$ $(2 \times 0,04 \text{ mL})$ Fertigungstoleranz) </p>				5								
<p>Messprotokoll: Aceton n-Hexan Alkohol Wasser Öl $m [\text{g}]$ $\rho = \frac{m}{V} [\text{g/cm}^3]$ $u_p = \left \frac{\partial p}{\partial m} \right u_m + \left \frac{\partial p}{\partial V} \right u_v$</p> <table border="1"> <tr> <td>Aceton</td> <td>19,76</td> <td>0,7904</td> <td>0,00260928</td> </tr> <tr> <td>n-Hexan</td> <td>16,43</td> <td>0,6572</td> <td>0,00218304</td> </tr> </table>				Aceton	19,76	0,7904	0,00260928	n-Hexan	16,43	0,6572	0,00218304	10
Aceton	19,76	0,7904	0,00260928									
n-Hexan	16,43	0,6572	0,00218304									
<p>Messprotokoll: Aceton: $\rho = 0,7904$ n-Hexan: $\rho = 0,6594$</p>				15								
<p>2) Dichtemessgerät: Einfüllen der Flüssigkeit in das U-Röhrchen mit einer Spritze über dafür vorgesehene Öffnung, Beleuchtung einschalten & überprüfen, ob keine Luftbläschen da sind (WICHTIG!) "F SOS" eingeben & Messung starten, Wert ablesen (Gerät vorher kalibrieren!) Nach Messung Röhrchen mit deion. Wasser spülen und via Pumpe trocknen unter Durchbeulen von Luft</p>				20								
<p>Messprotokoll: Aceton: $\rho = 1,35867$ n-Hexan: $\rho = 1,37506$</p>				25								
<p>3) schwarze Abdeckklappe öffnen & Probenblock abwischen mit Linsentuch: Nicht Gegenstände zerkratzen! So viel der zu messenden Flüssigkeit darauf, bis runde Fläche vollkommen bedeckt. Deckel schließen, START drücken, Wert ablesen, wieder abwischen</p>				30								
<p>Auswertung: $u_p = \left \frac{\partial p}{\partial m} \right u_m + \left \frac{\partial p}{\partial V} \right u_v = \frac{1}{V} \cdot u_m + \left -\frac{m}{V^2} \right \cdot u_v = \frac{u_m}{V} + \frac{m \cdot u_v}{V^2}$ (Abwegmessung)</p>				35								
<p>Bsp. mit $u_m \approx \pm 1 \text{ mg}$ Messunsicherh. der Waage $\rightarrow u_m = 0,002 \text{ g}$ $u_v \Rightarrow$ Fertigungstoleranz v. 25 mL Messkolben $= \pm 0,04 \text{ mL} \rightarrow u_v = 0,08 \text{ mL}$ $\rightarrow u_p = 0,00260928 \approx 0,0026 \text{ g/mL} \rightarrow \rho_{\text{act.}} = 0,7904(26)$ $\rho_{\text{literatur}} = 0,6572(28)$</p>				40								
<p style="text-align: right;">22 Literaturwerte:</p>				45								
SIGNATURE	DATE	WITNESS	DATE	46								
Continued to Page				47								

BOOK	4	PAGE
TITLE	PROJECT	
DATE 5.1.2024		
<i>Continued from Page</i>		
MESSUNG & ABLW. V. DÄR-DATEN		
5 → Bestimmung versch. Siededruck - Siedetemp. Paare $\{p_i, T_i\}$ von Aceton & n-Hexan.		
<u>Messgeräte:</u> <ul style="list-style-type: none"> • 2-Hals holben • Dimrothkühler • regelbar. elektr. Heizung (WINMLER WTLG 2) Laborheizplatte mit KETT WL=10 Heizwandler • GREISINGER GMH 3210 Digital-Temp. Messgerät (0.1K Auflösung) • Membranvakuumpumpe (BÜCHI VAC V-503) (Endvakuum 10 mbar mögl.) • Vakuumkontroller (BLÜCHI 1-100) • 25L Ballantank (Stahlkonsche) 		
<u>Aufbau:</u>		
<u>Dimrothkühler</u>		
<i>Continued to Page</i>		
SIGNATURE	DATE	WITNESS