

Figur 1.1. Spredning og målesikkerhed.

Fraktil	97,5 % = 0,975	99,5 % = 0,995	99,95 % = 0,9995
$f = 1$	12,71	63,66	636,6
$f = 2$	4,30	9,93	31,6
$f = 3$	3,18	5,84	12,9
$f = 4$	2,78	4,60	8,61
$f = 5$	2,57	4,03	6,87
$f = 6$	2,45	3,71	5,96
$f = 7$	2,37	3,50	5,41
$f = 8$	2,31	3,36	5,04

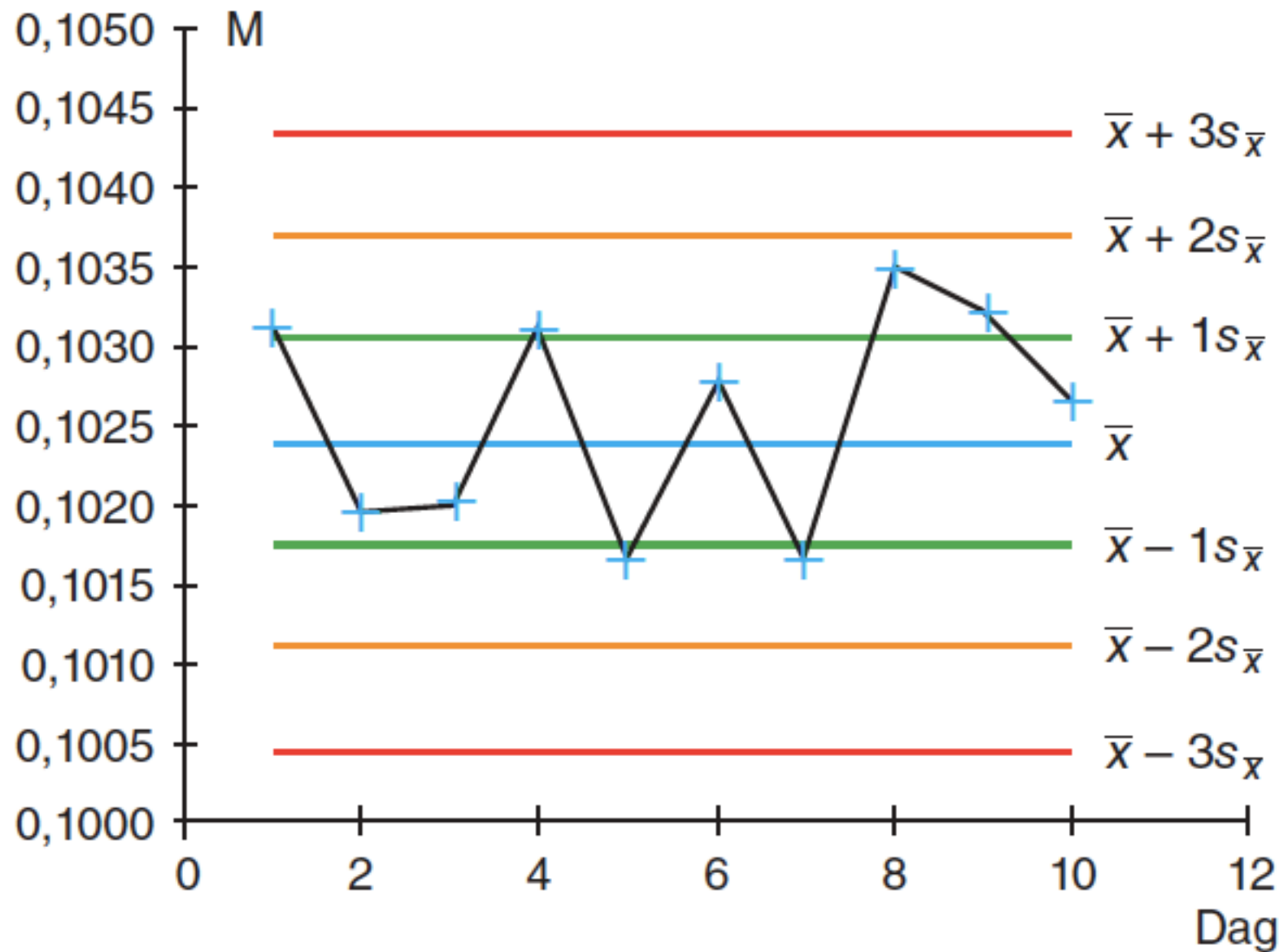
Tabel 1.1. Uddrag af t -tabel.

Konfidensniveau	Fraktil	Signifikansniveau (α)	p -værdi
95 %	97,5 %	5 %	0,05
99 %	99,5 %	1 %	0,01
99,9 %	99,95 %	0,1 %	0,001

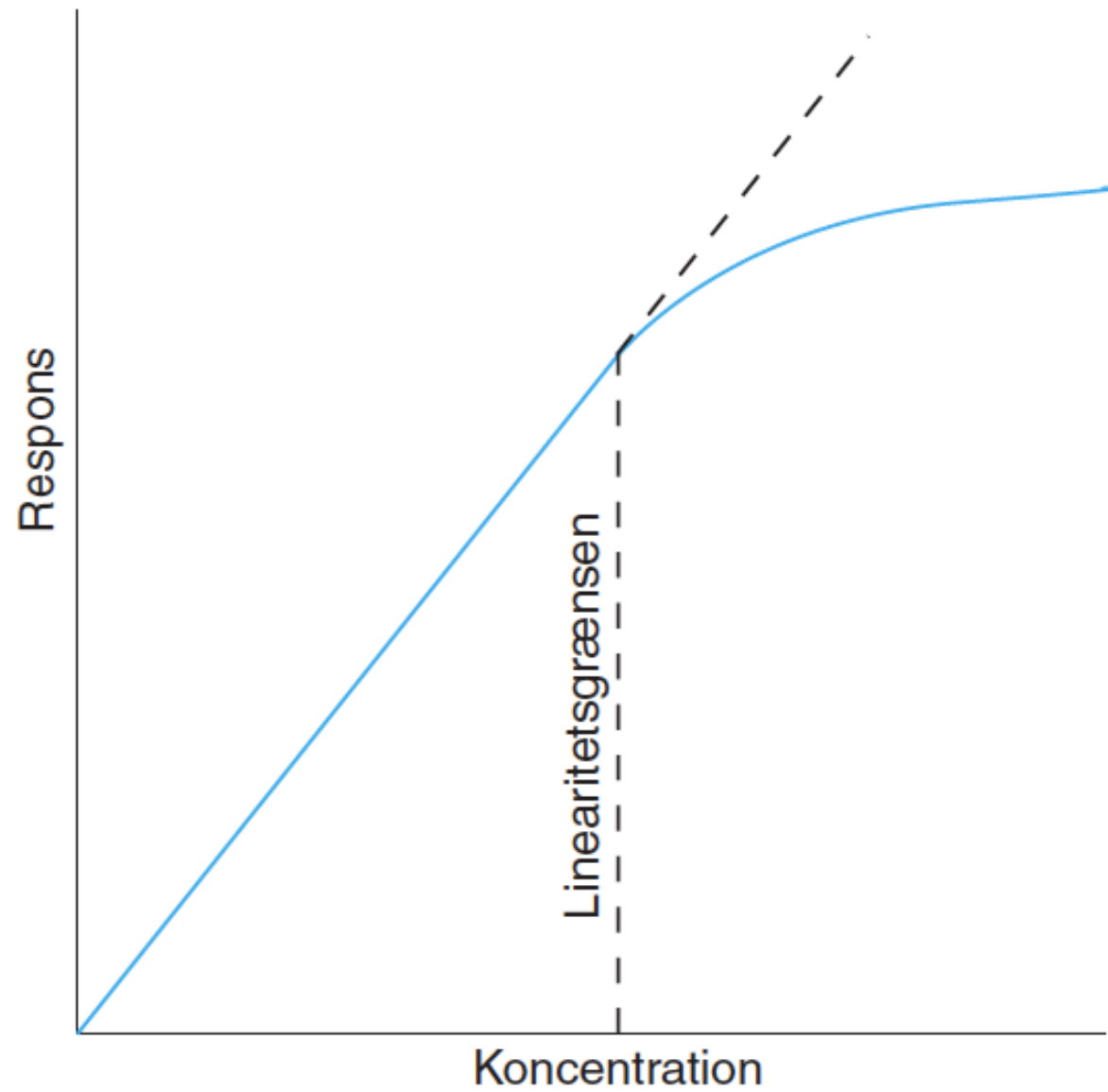
Tabel 1.2.

Antal målinger	95 %-konfidensniveau
3	0,970
4	0,829
5	0,710
6	0,625
7	0,568

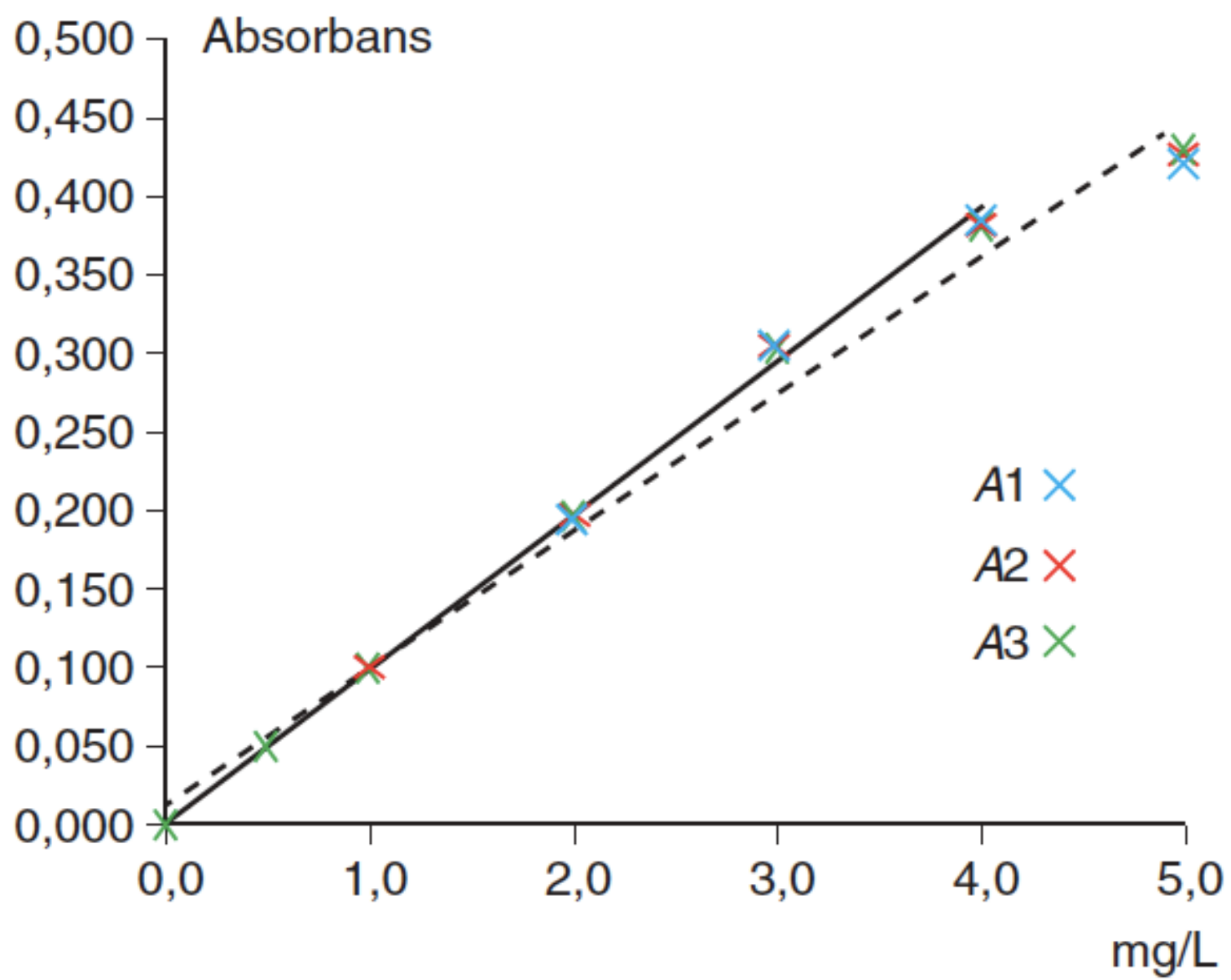
Tabel 1.3. *Q*-test-tabel



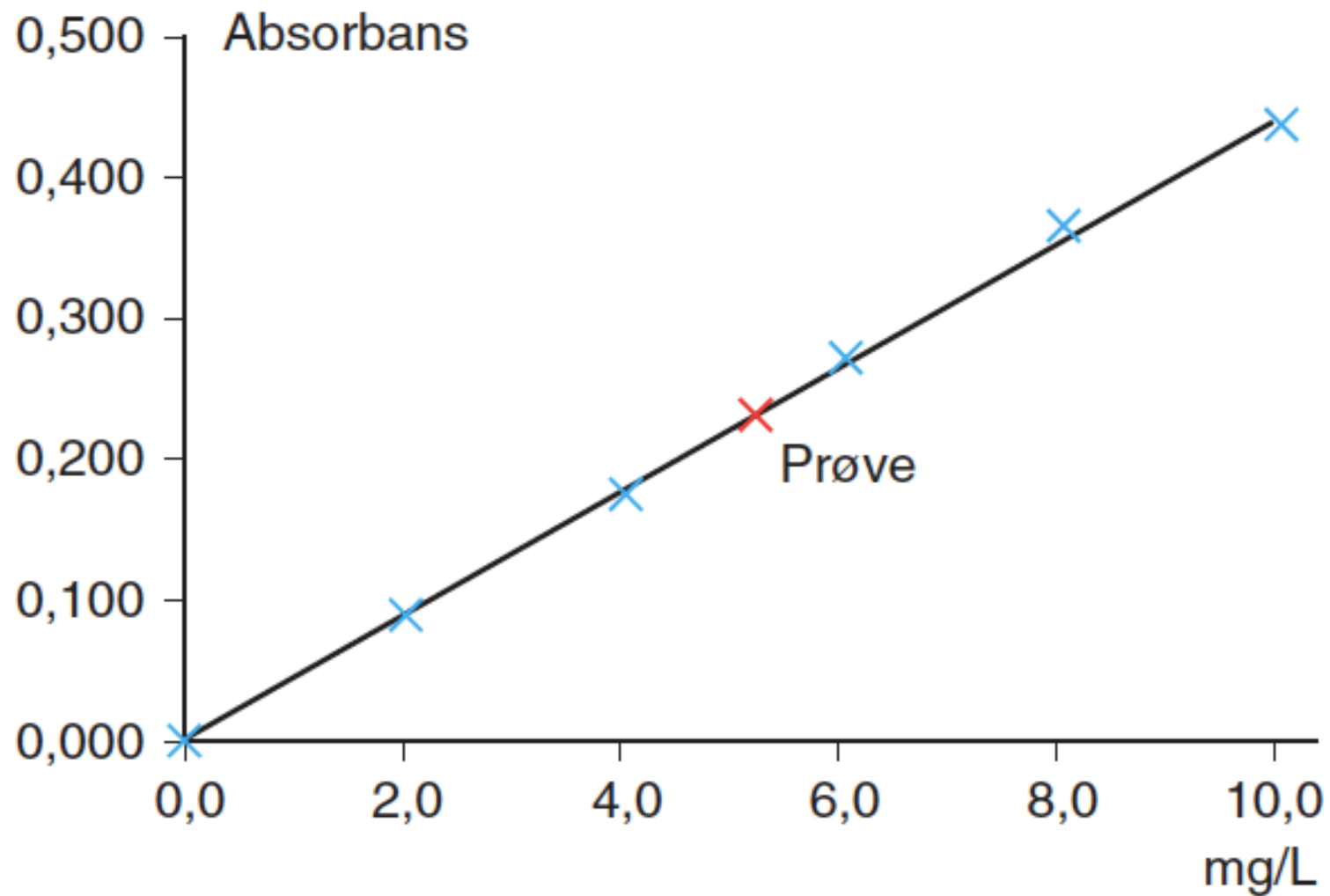
Figur 1.2. \bar{x} -kontrolkort.



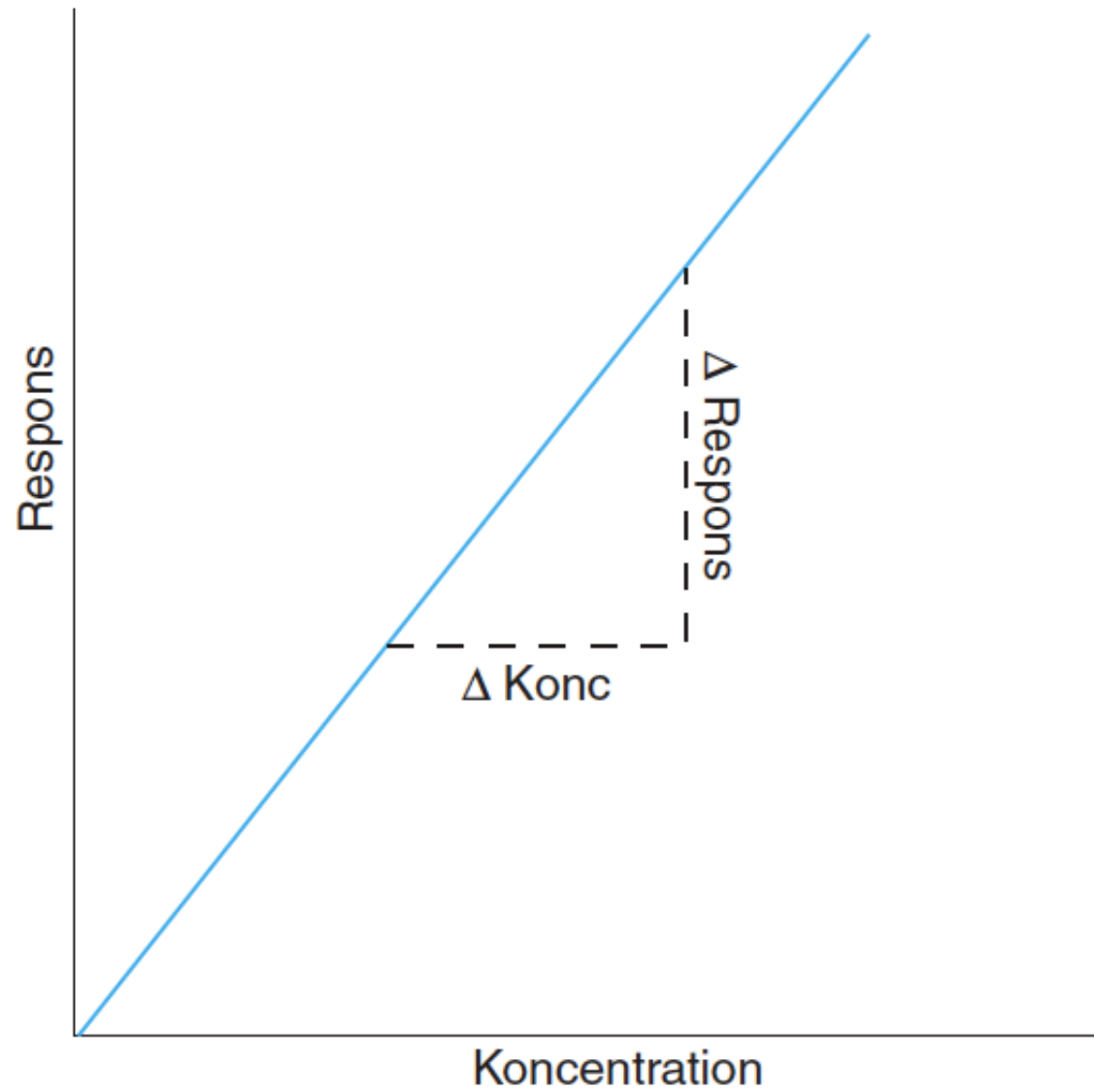
Figur 2.1. Standardkurve og linearitetsgrænse.



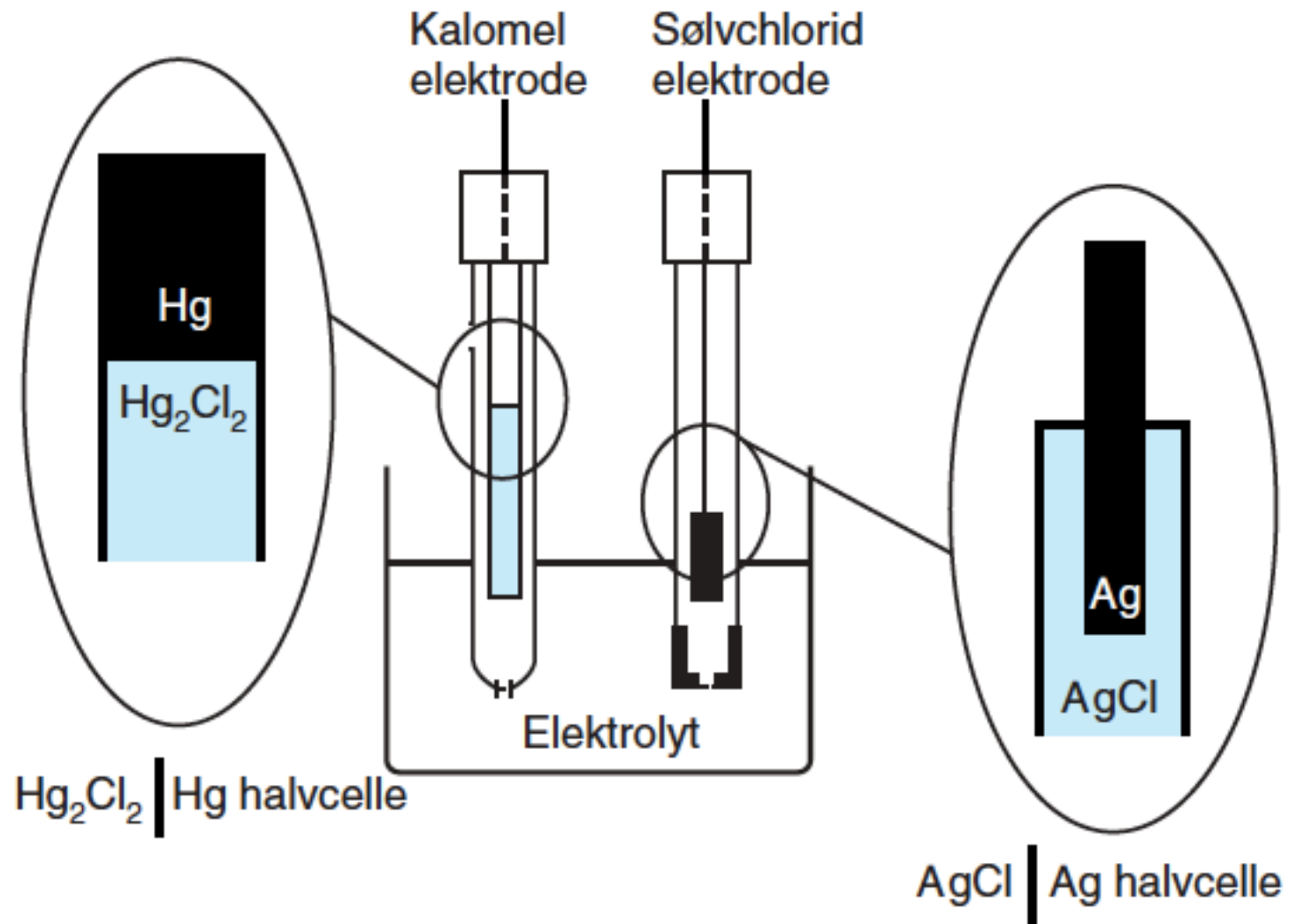
Figur 2.2. Standardkurve for fosfat.



Figur 2.3. Standardkurve for spektrofotometrisk bestemmelse af jern



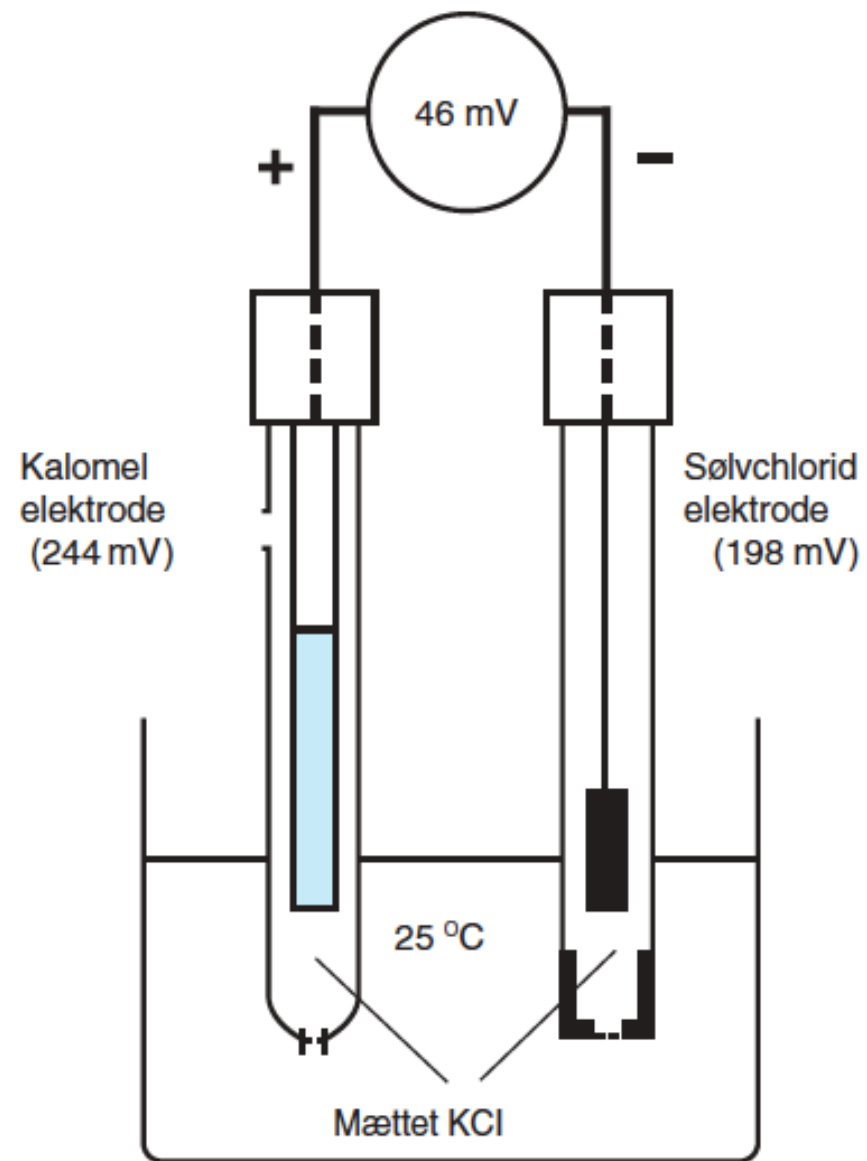
Figur 2.4. Følsomhedsbestemmelse.



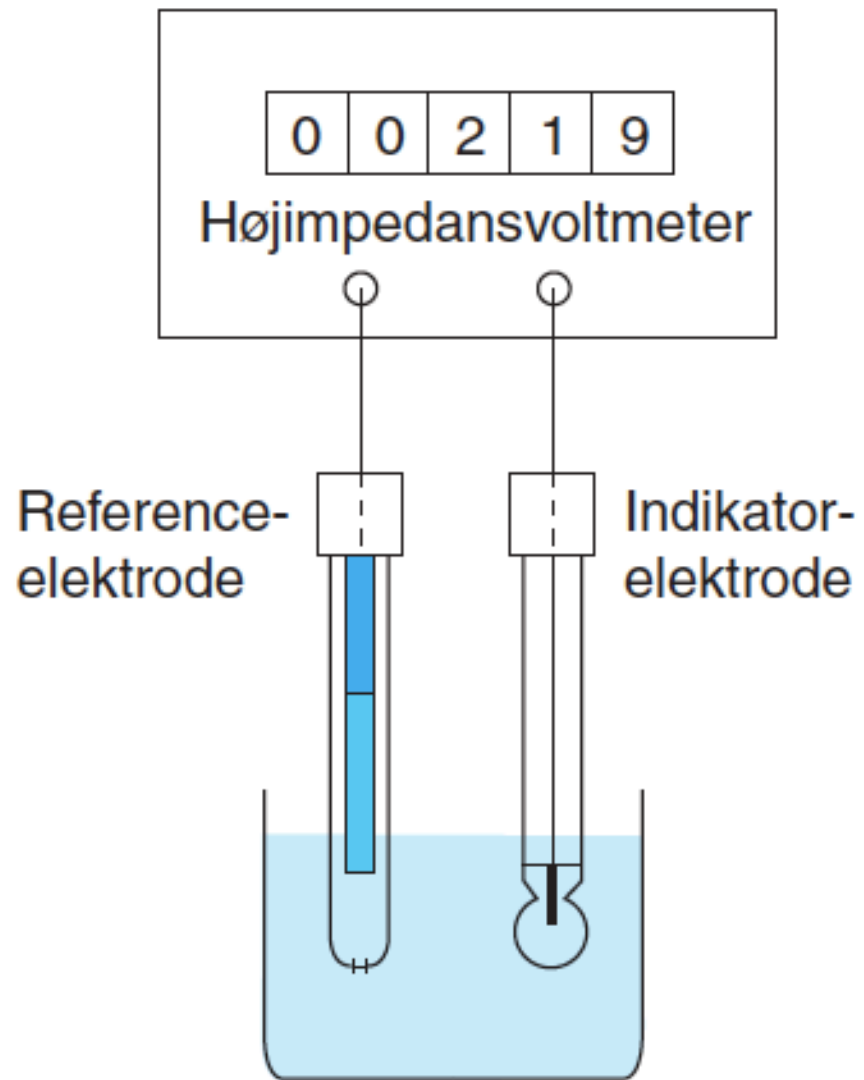
Figur 3.1. Måleopstilling med to elektroder.

Elektrode	E_0 mV ²¹ (25 °C)
Kalomel	+244
Sølvchlorid	+198

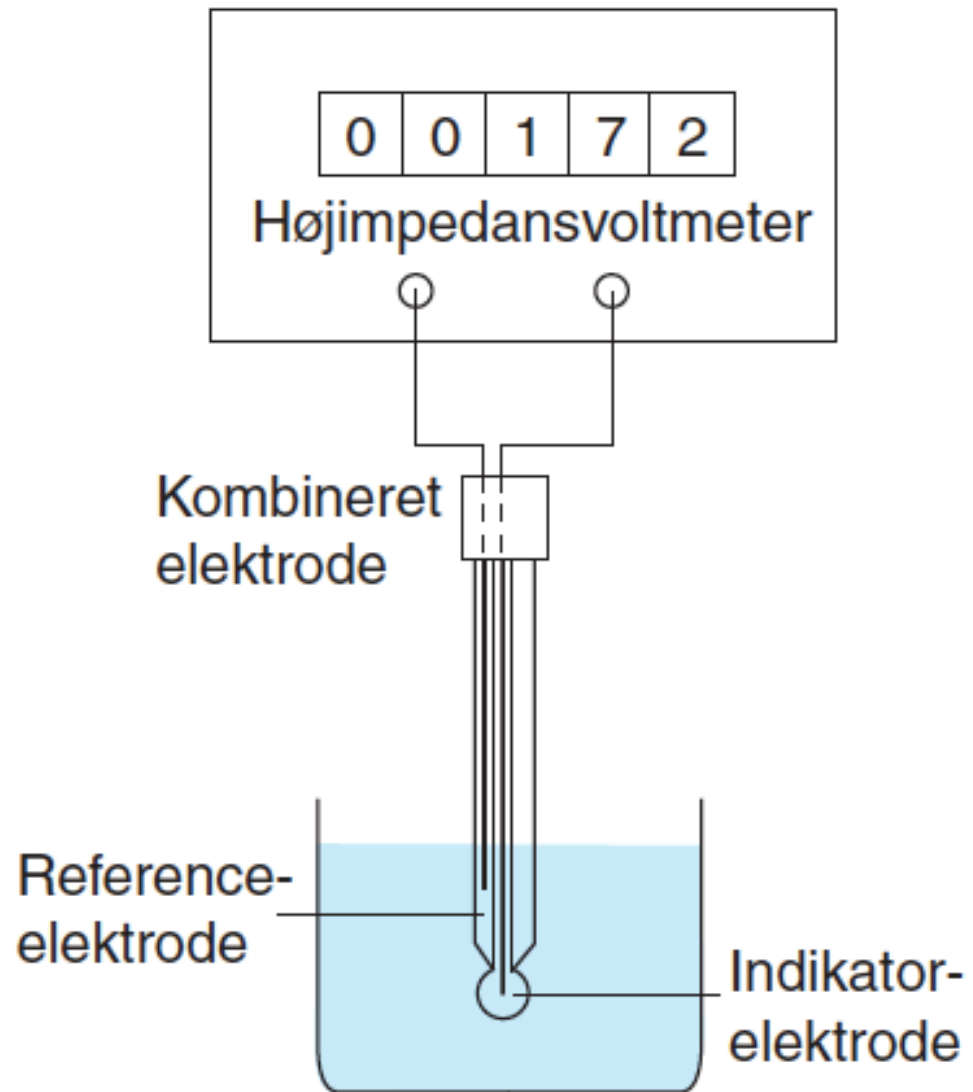
Tabel 3.1.



Figur 3.2. Potentialemåling mellem to elektroder

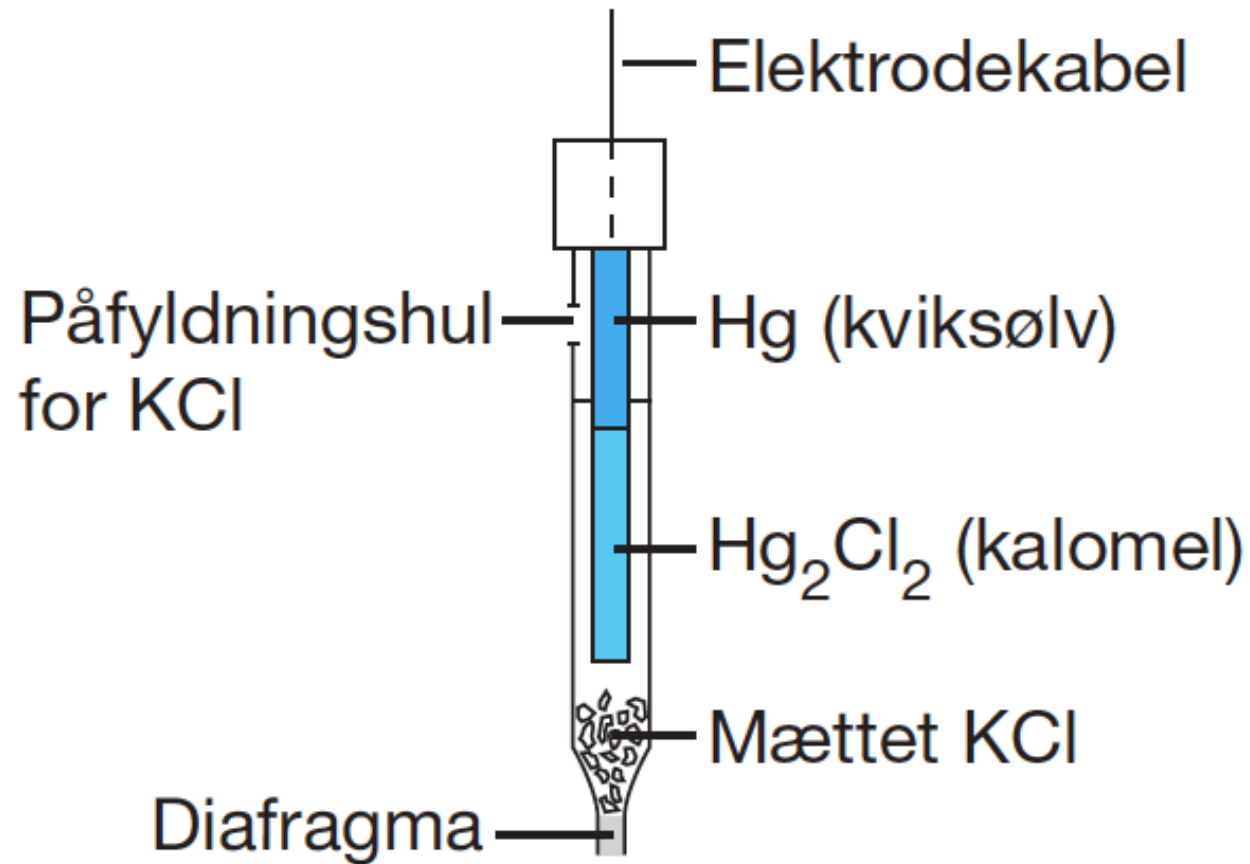


Figur 3.3. Måling med to elektroder.



Figur 3.4. Måling med kombielektrode.

Kalomelreferenceelektrode

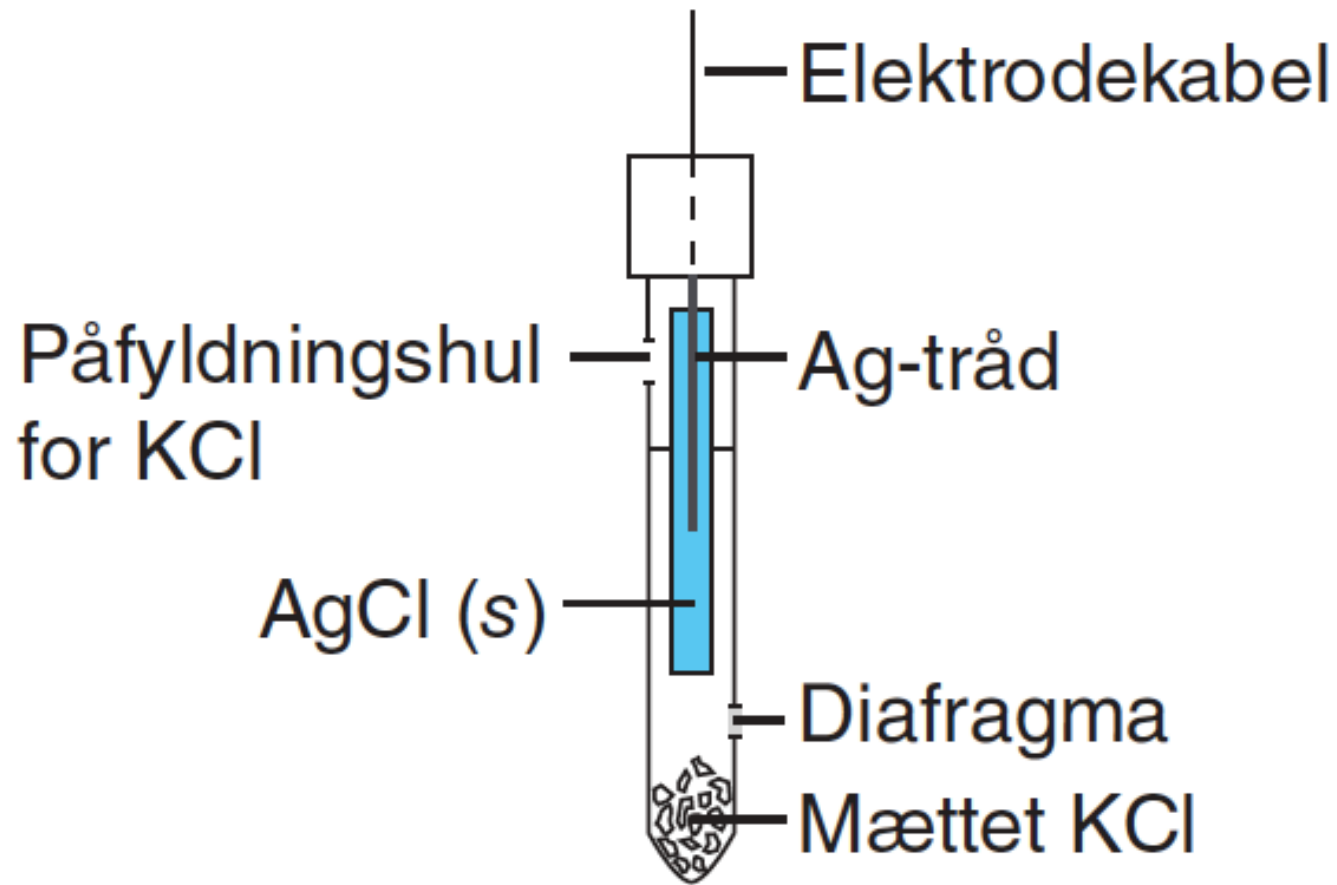


Figur 3.5. Kalomelelektroden.

°C	0	25	40	60
mV	260	244	234	220

Tabel 3.3. ²⁹

AgCl-referenceelektrode



Figur 3.6. Sølvchloridelektroden.

°C	0	25	40	60	80	95
mV	220	197	181	160	138	121

Tabel 3.4.

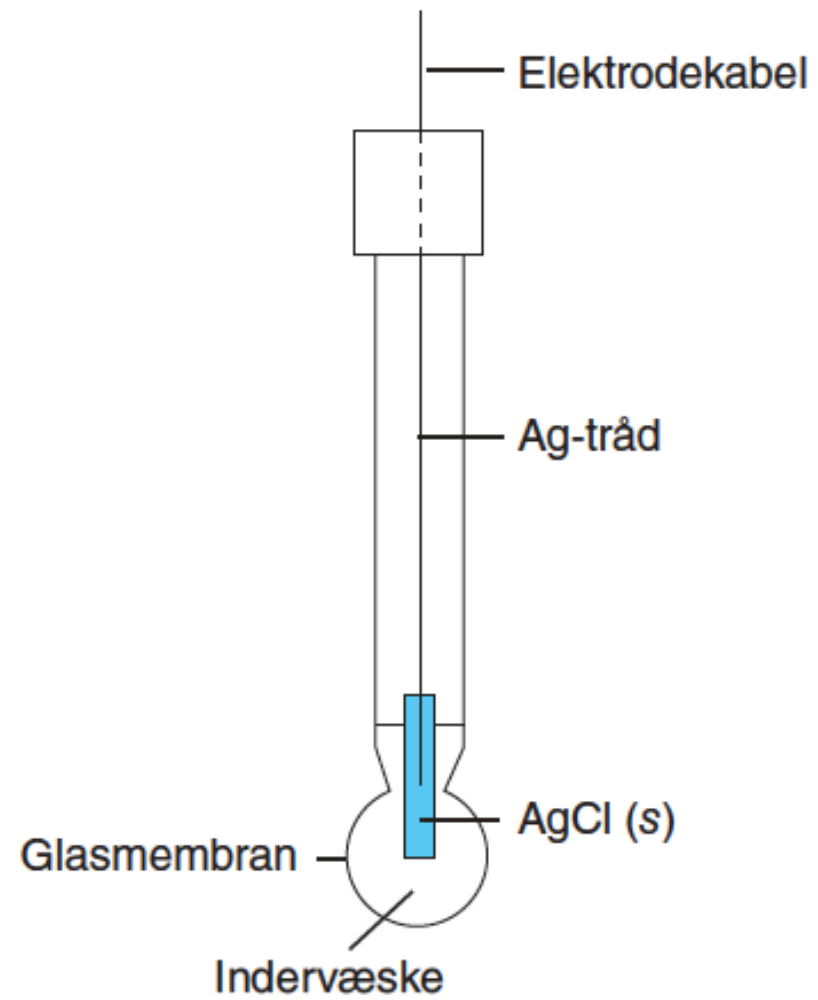
<i>Kation</i>	<i>Mobilitet</i>	<i>Anion</i>	<i>Mobilitet</i>
Li^+	39	OH^-	198
Na^+	50	SO_4^-	80
K^+	74	Br^-	78
NH_4^+	74	Cl^-	76
H^+	350	NO_3^-	72

Tabel 3.5.

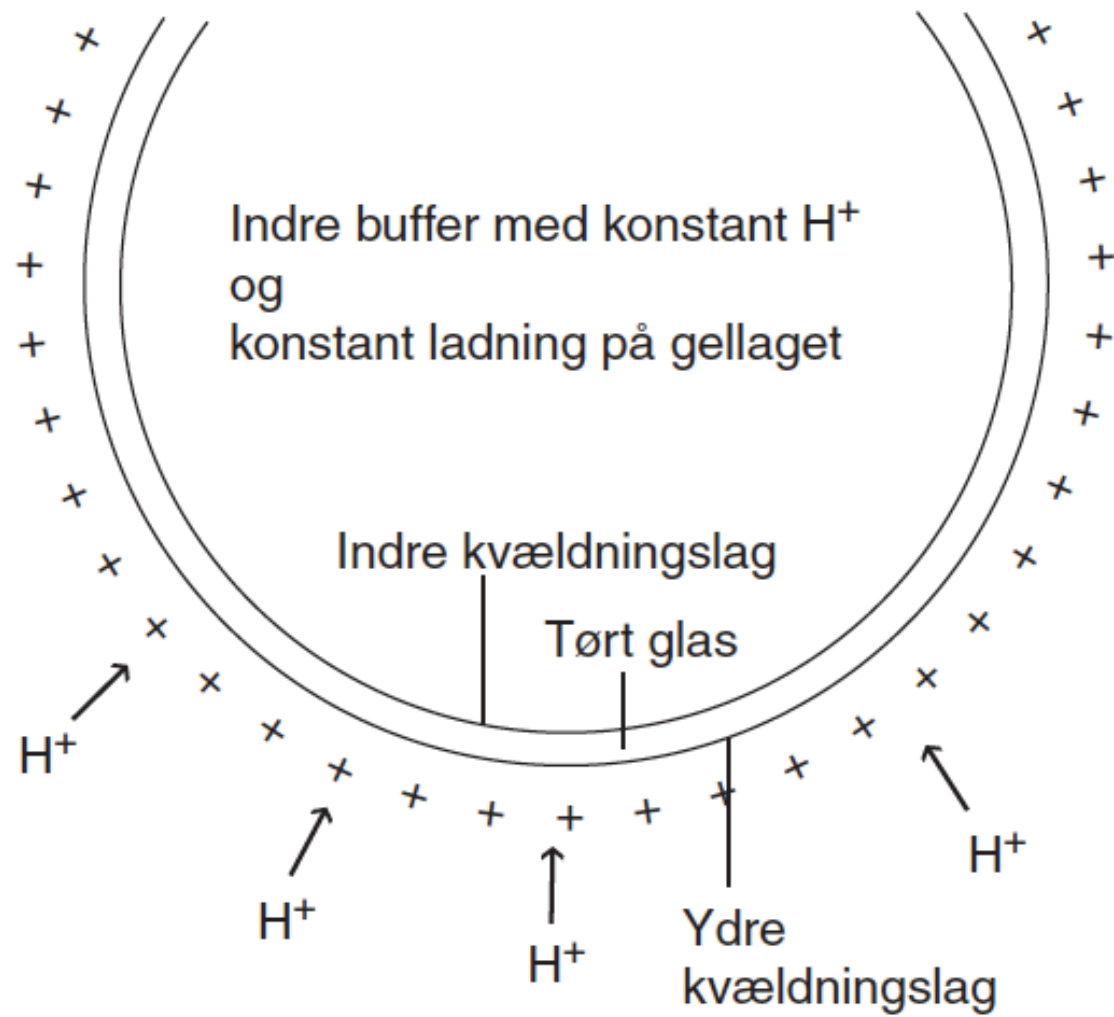
<i>Opløsning (molaritet)</i>	<i>Væskebropotentiale (mV)</i>
HCl (1 M)	14,1
HCl (0,1M)	4,6
HCl (0,01M)	3,0
KCl (0,1M)	1,8
NaOH (0,01M)	2,3
NaOH (0,1M)	-0,4
NaOH (1M)	-8,6

Tabel 3.6.

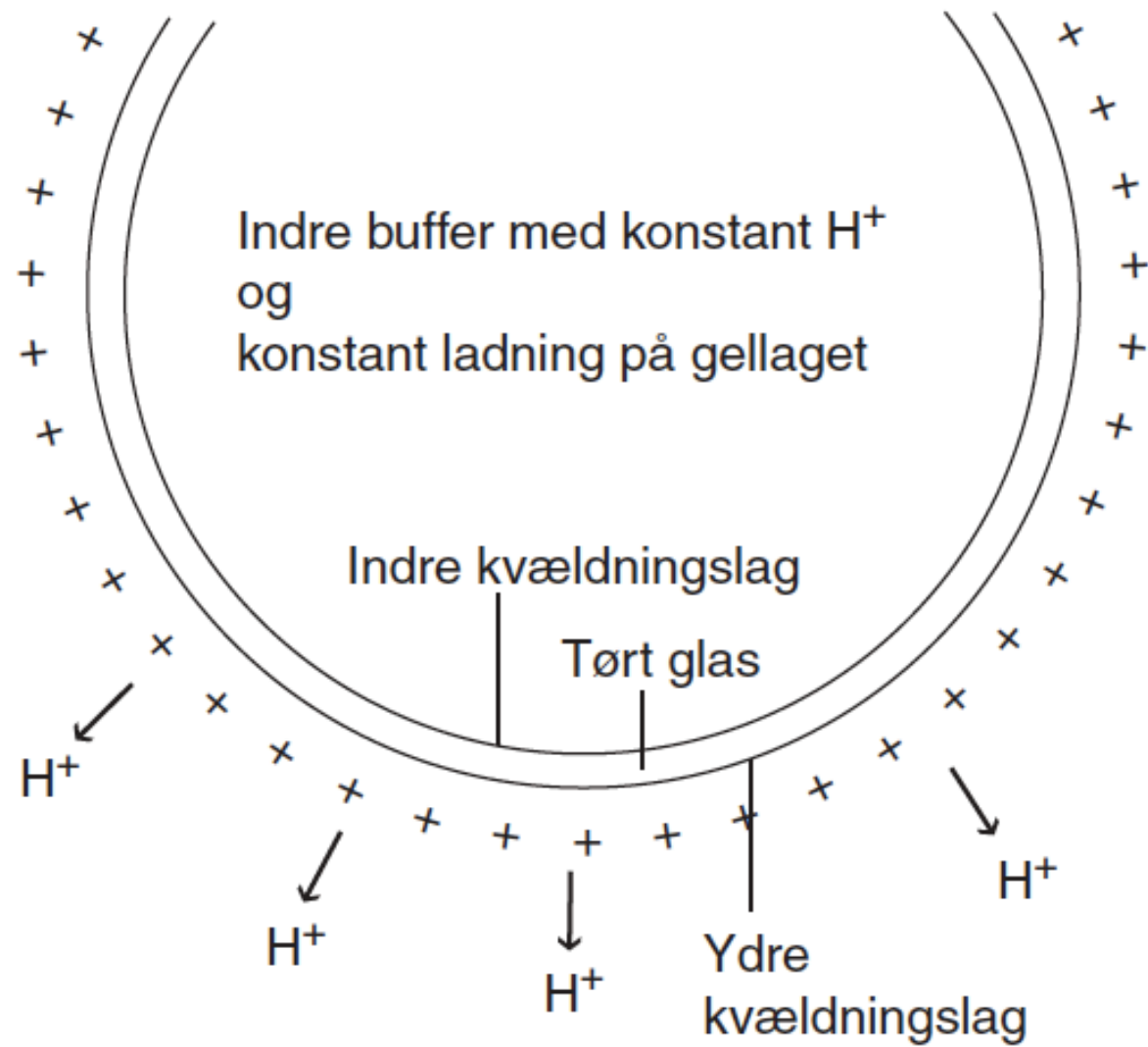
Glaselektrode



Figur 3.7. Glaselektroden.

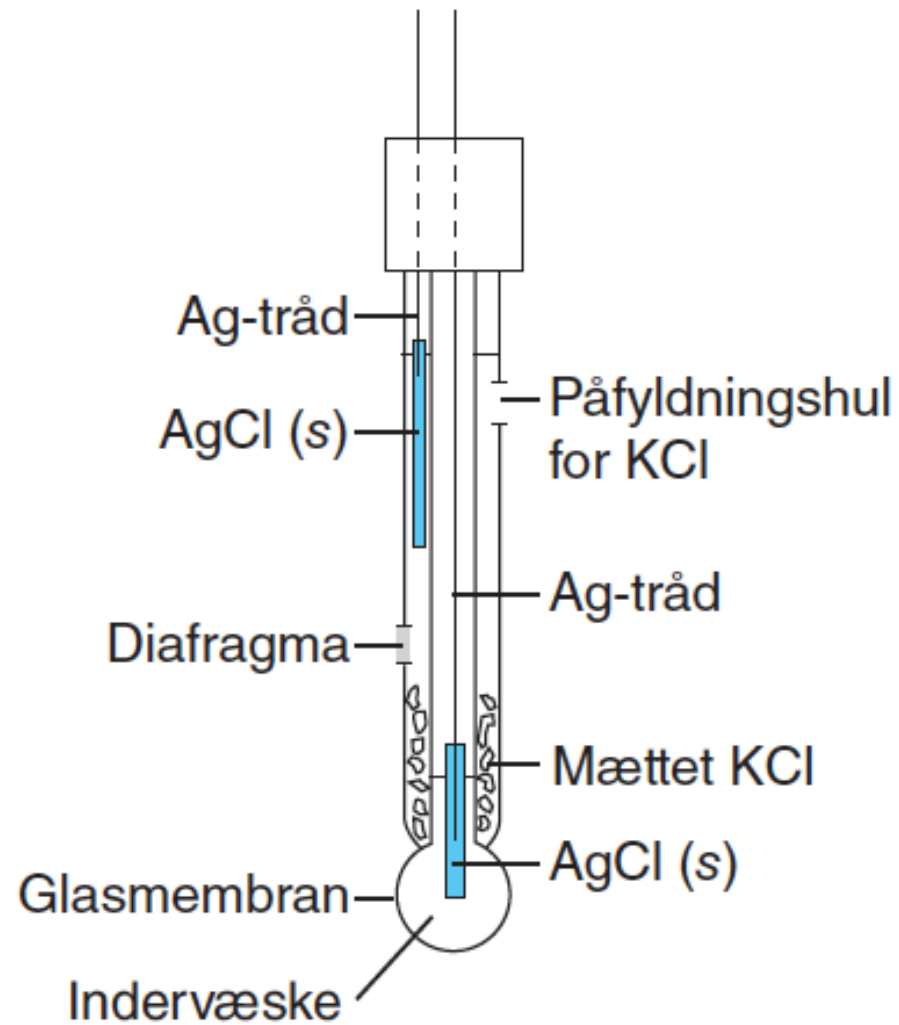


Figur 3.8. Glasmembranens overflade ved neddykning i sur væske.³³



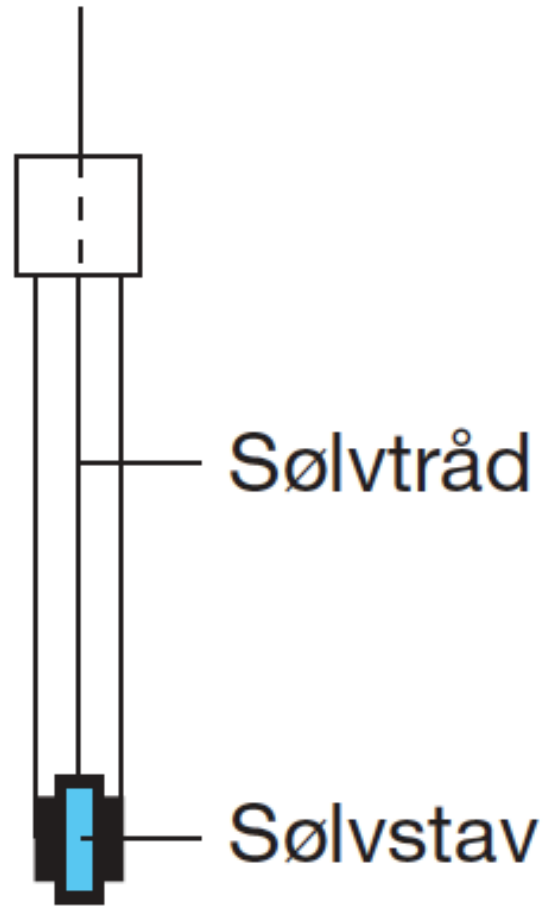
Figur 3.9. Glasmembranens overflade ved neddykning i basisk væske.

Kombineret pH-elektrode



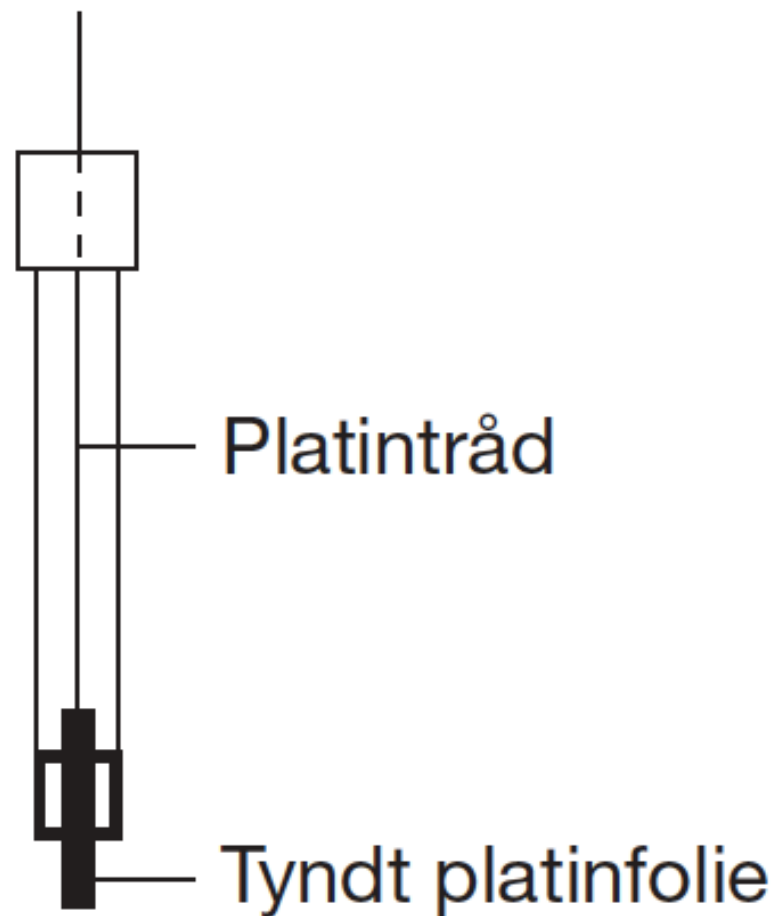
Figur 3.10. Kombielektrode til pH-måling.

Sølvelektrode



Figur 3.11. Sølvelektrodens opbygning.

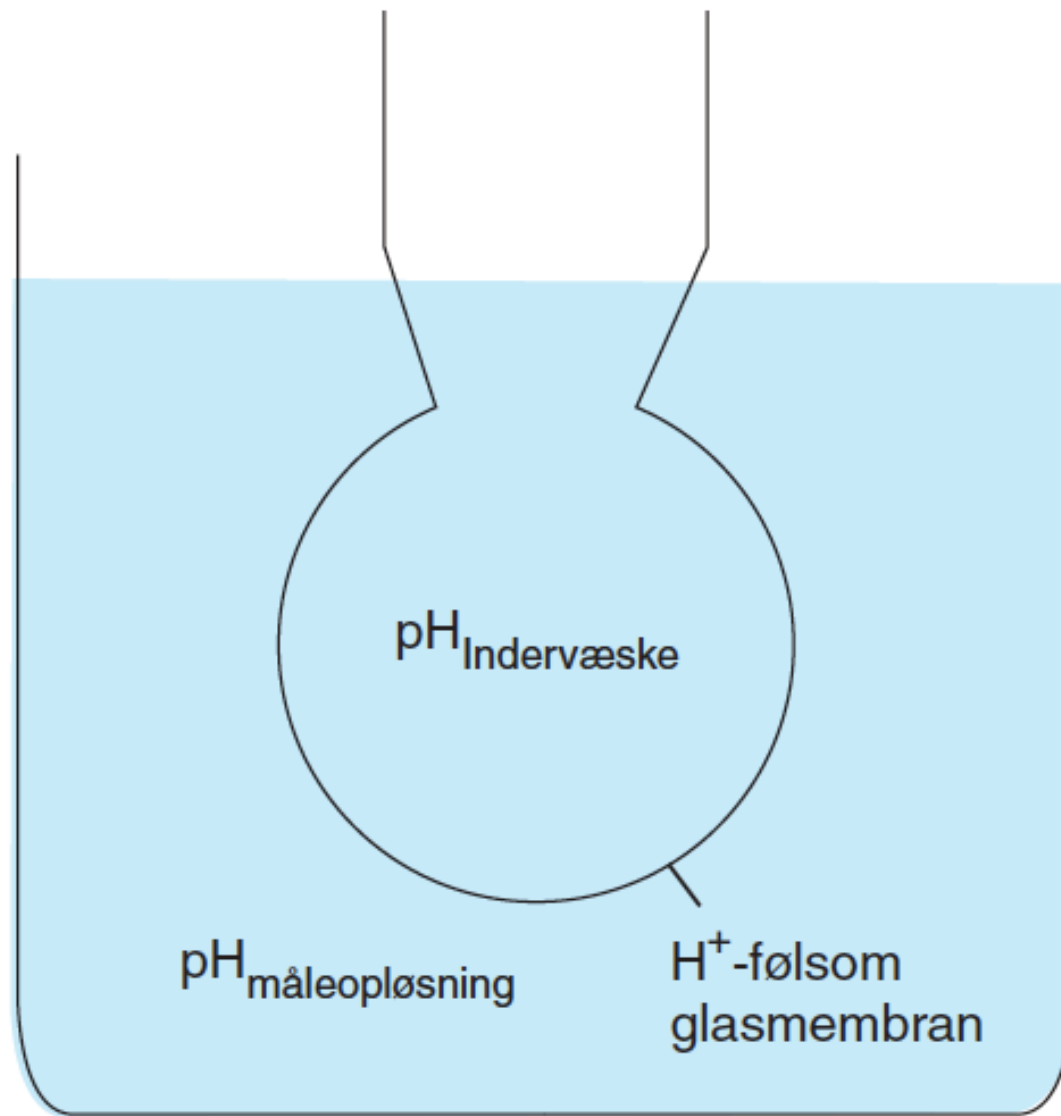
Platinelektrode



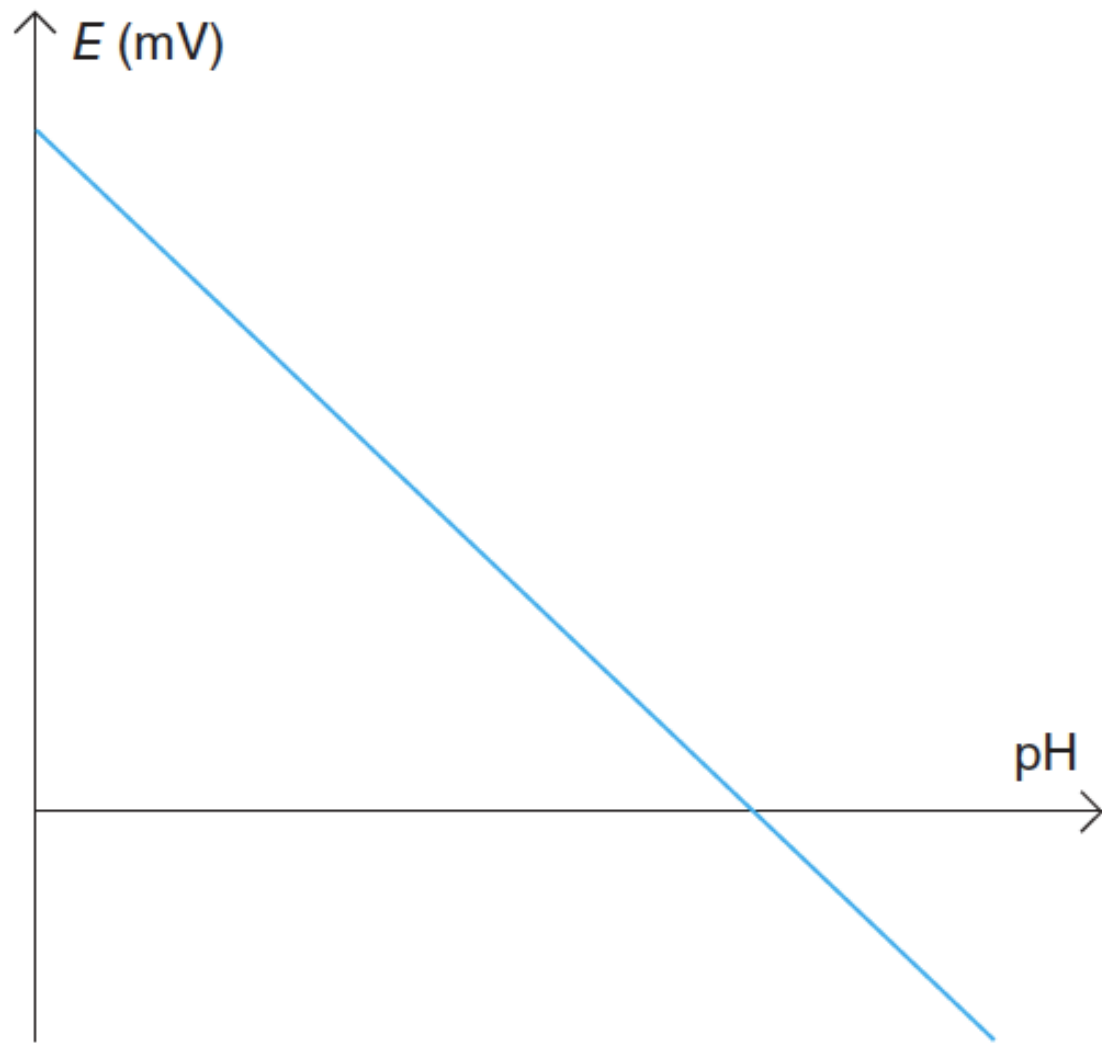
Figur 3.12. Platinelektrodens opbygning.

l (M)	0,001	0,005	0,01	0,05	0,1
γ	0,967	0,935	0,915	0,857	0,830

Tabel 3.7.



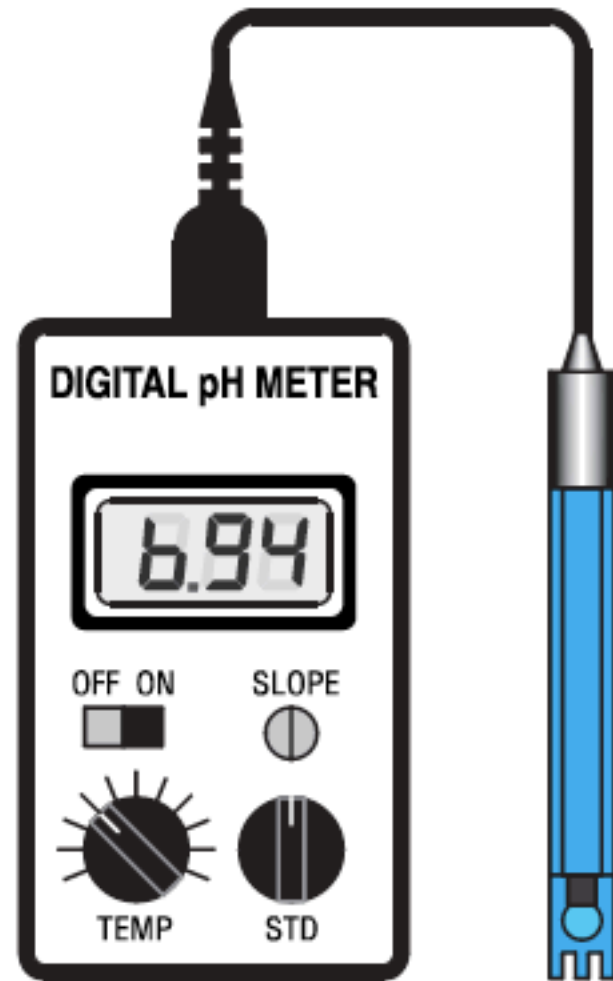
Figur 3.13. Glaselektroden neddyppet i måleopløsning.



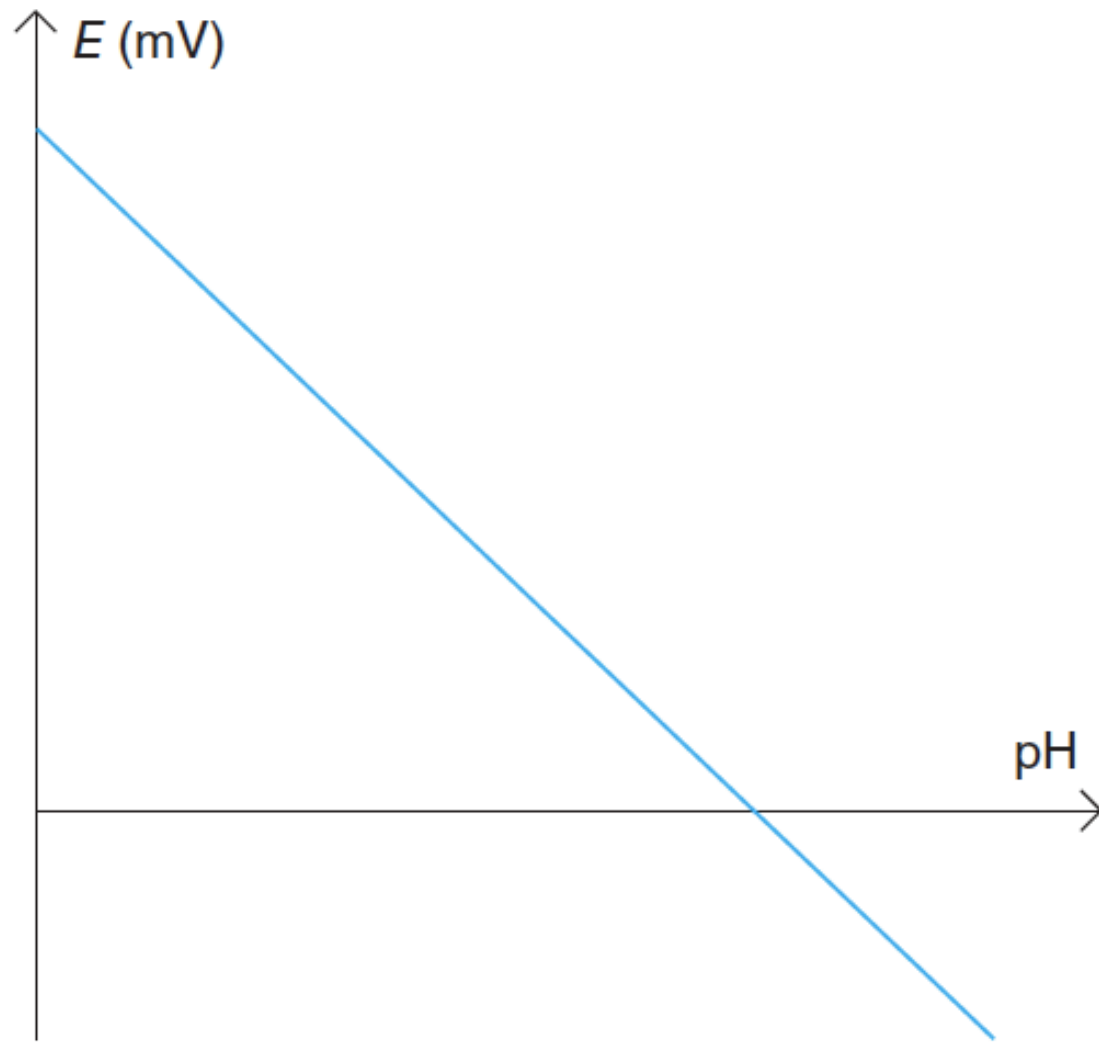
Figur 3.14. Lineær sammenhæng mellem pH og elektrodepotentialet E .



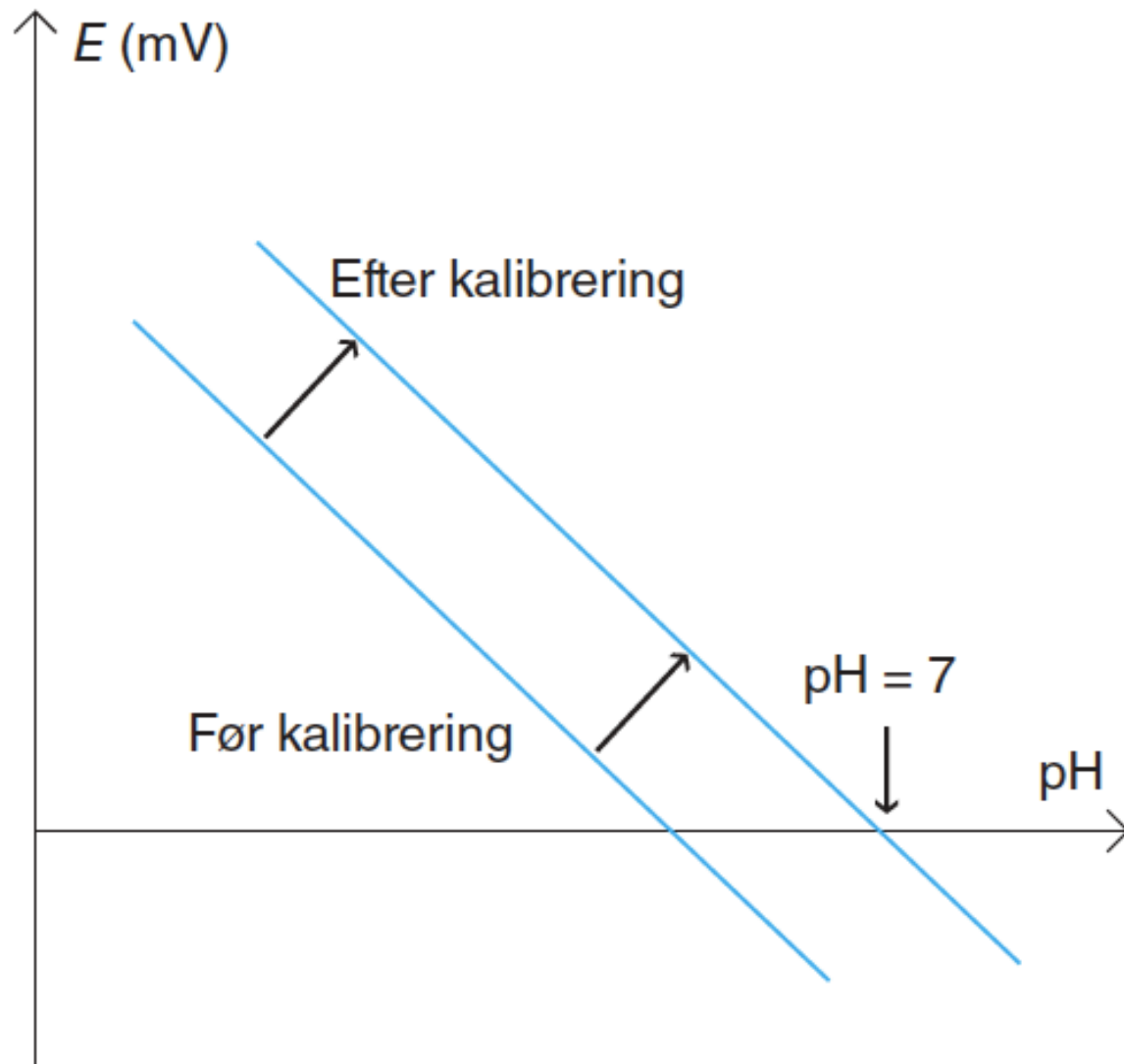
Figur 3.15. Moderne, stationært pH-meter.



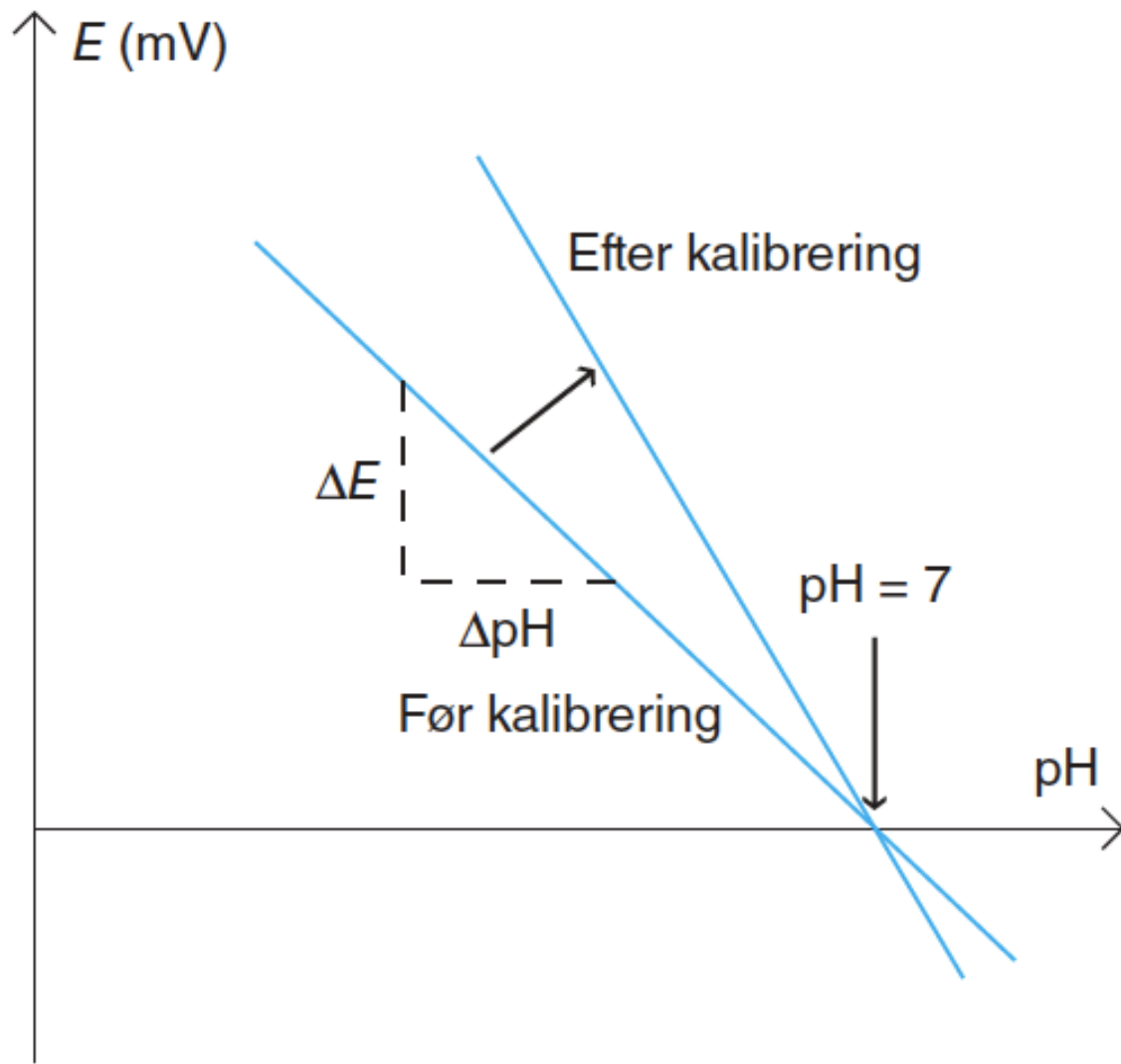
Figur 3.16. Transportabelt pH-meter.



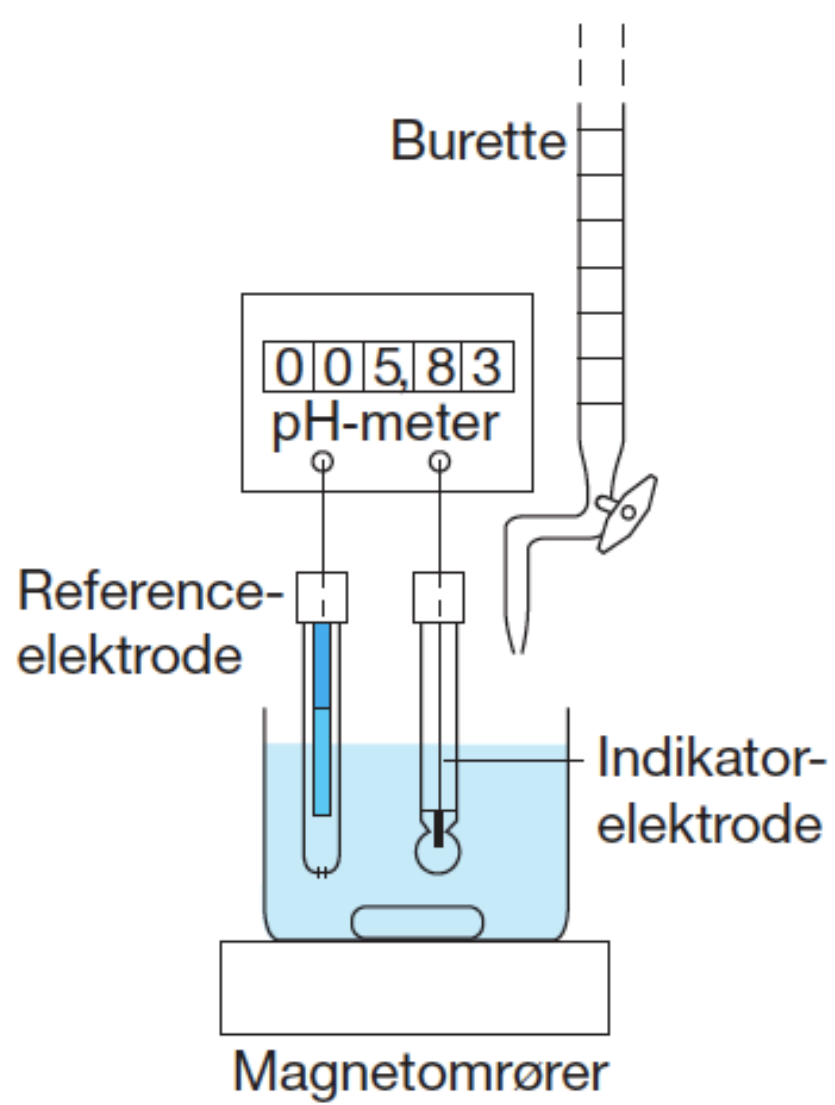
Figur 3.17. Teoretisk afbildning af Nernst-ligningen for pH-elektroder.



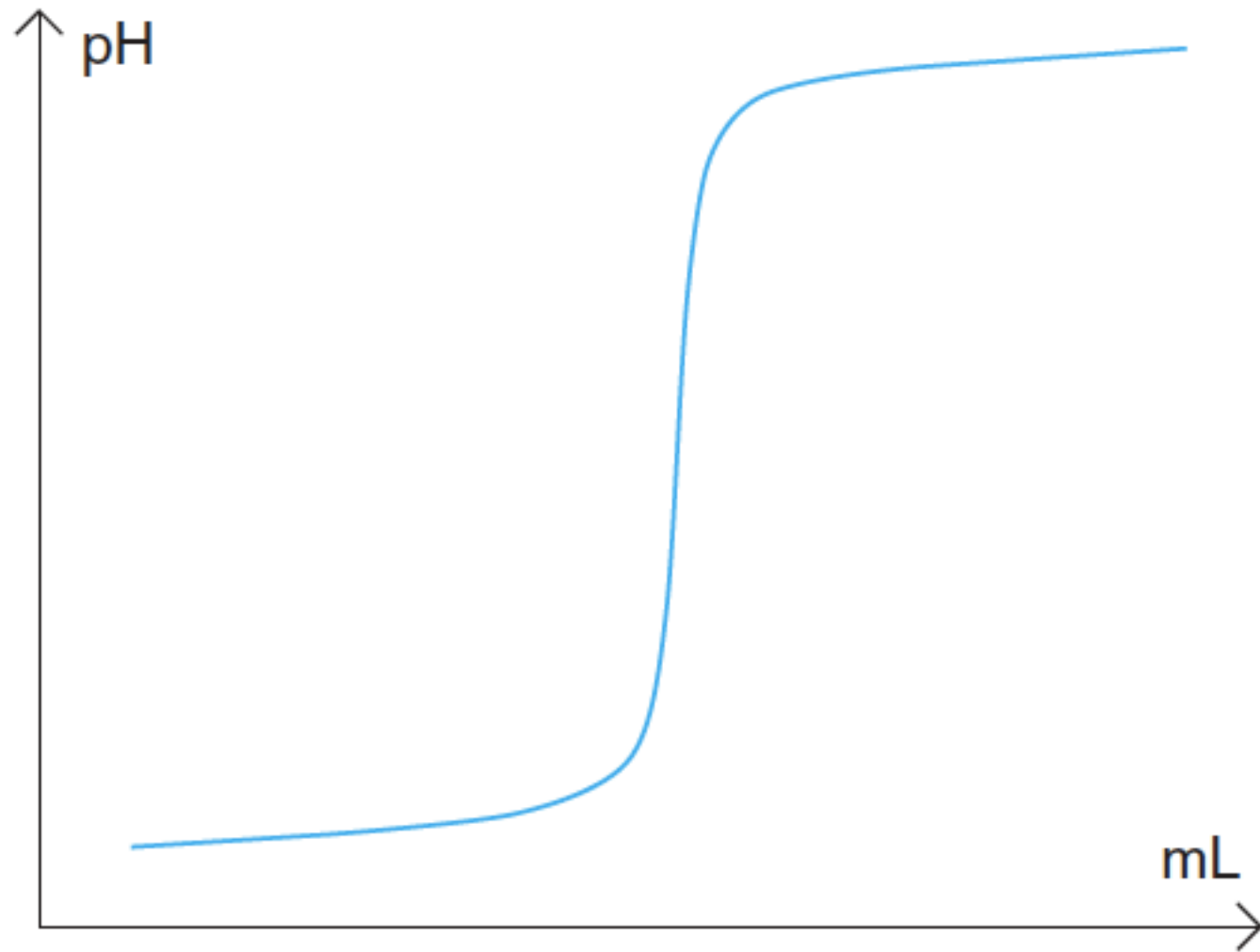
Figur 3.18. Toppunktskalibrering, første buffer med pH 7.



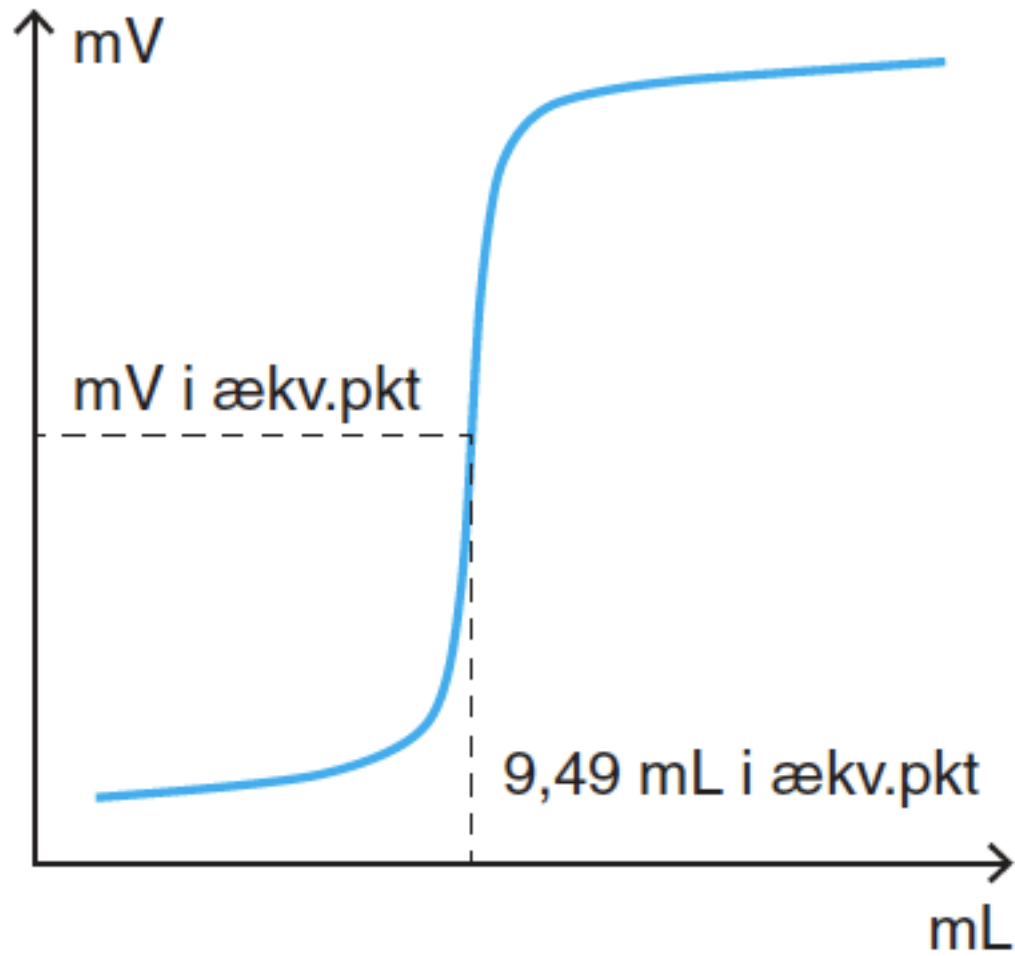
Figur 3.19. Toppunktskalibrering, anden buffer med pH 4 eller 10.



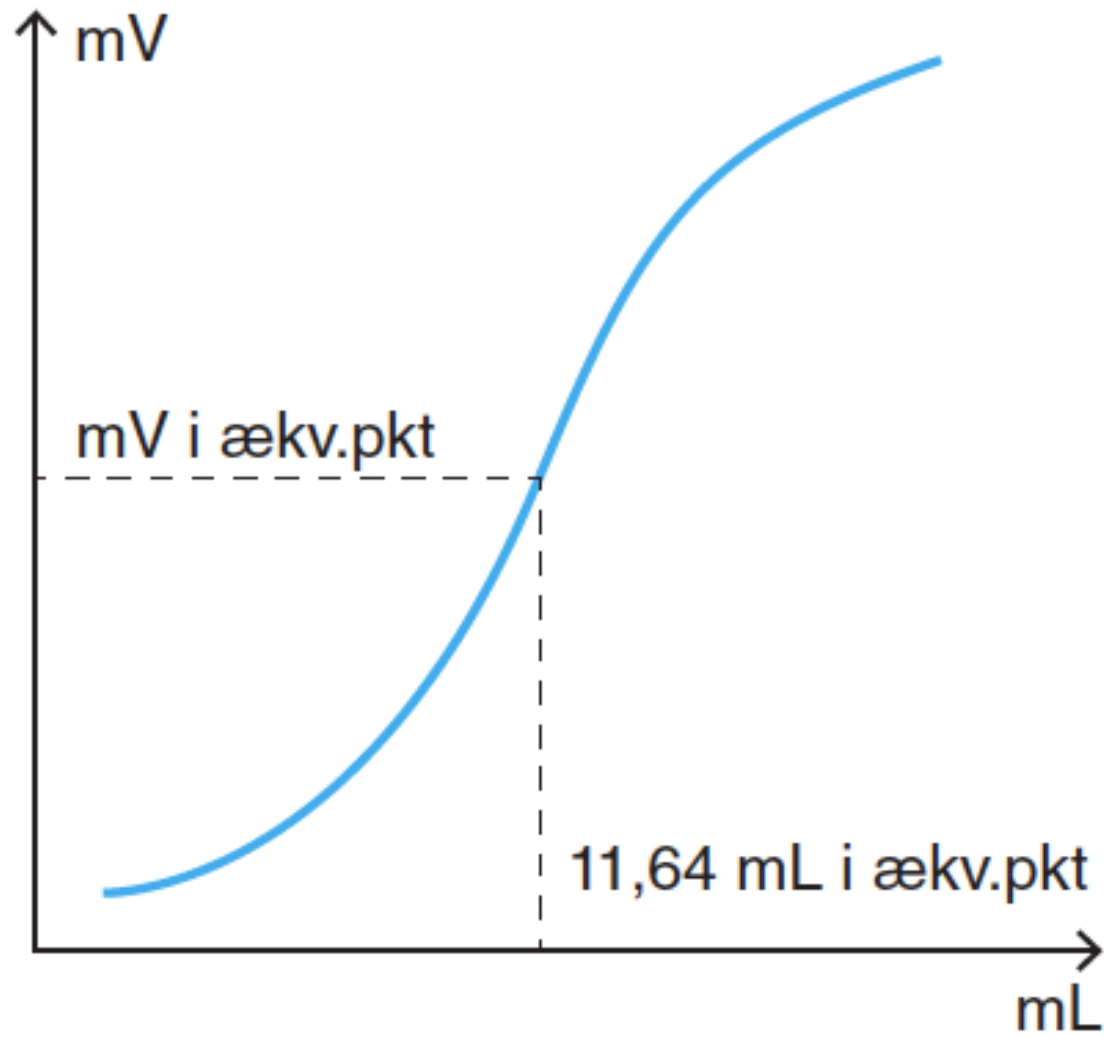
Figur 3.20. Opstilling til simpel potentiometrisk titrering.



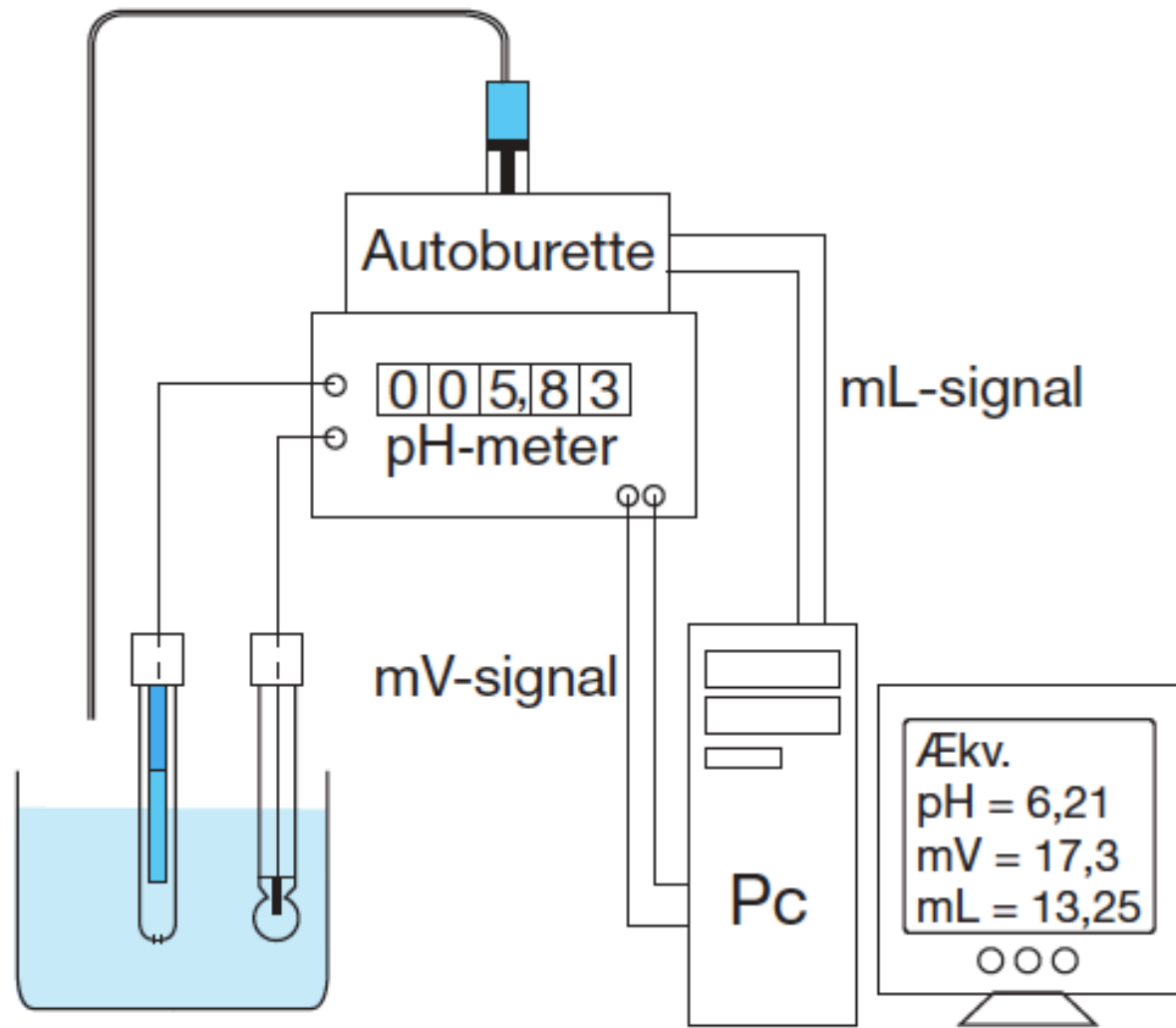
Figur 3.21. Titrerkurve fra potentiometrisk syre/base-titrering.



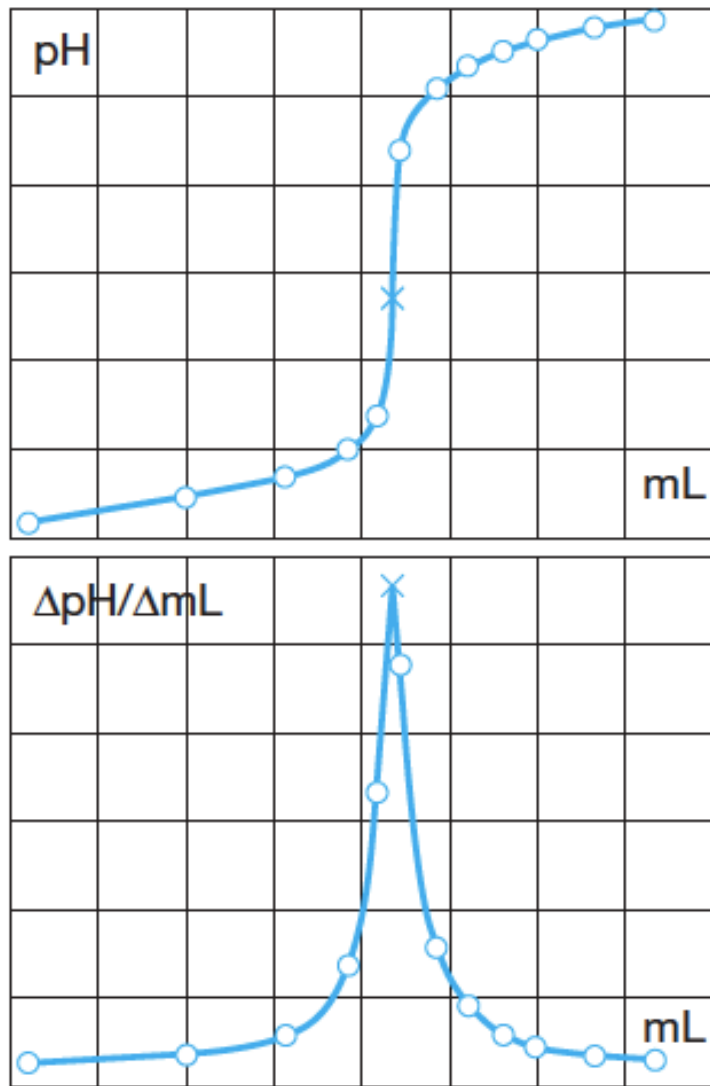
Figur 3.22. Titreringskurve med omslagspunkt.



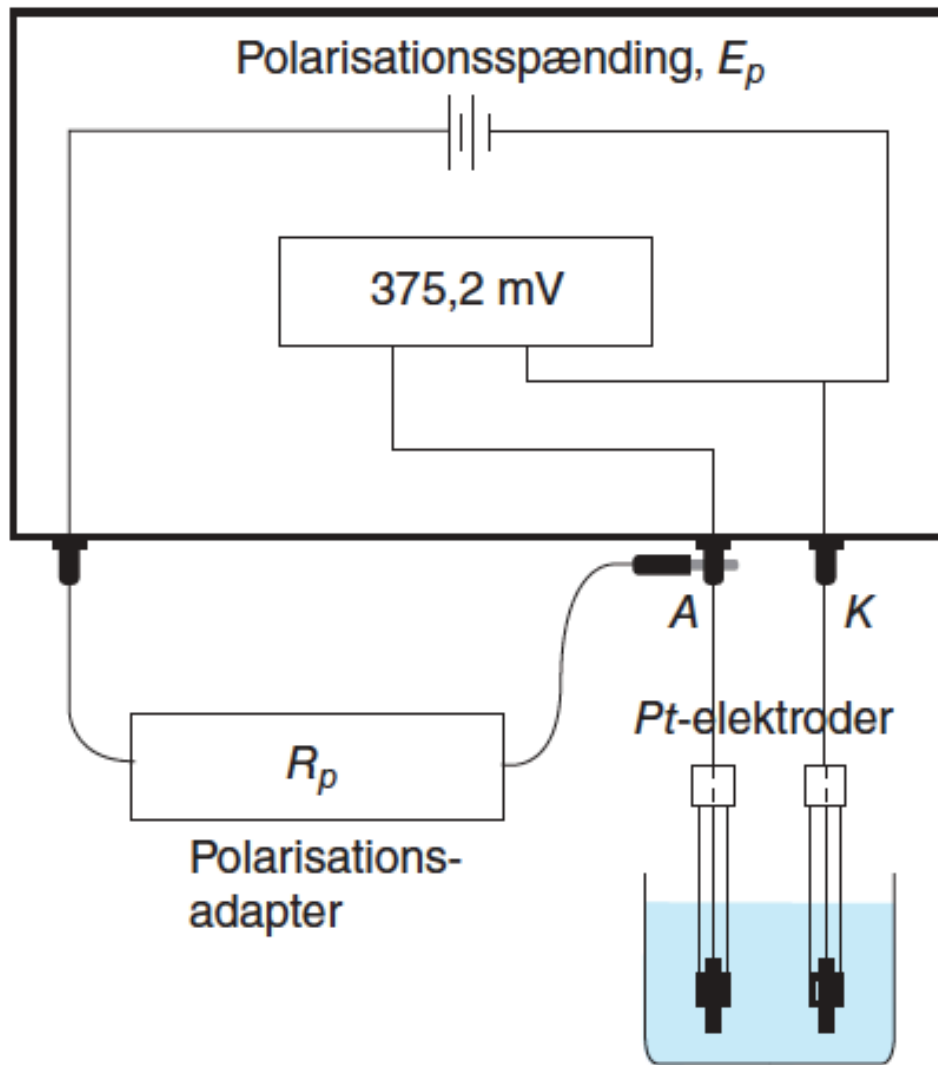
Figur 3.23. Titreringskurve med ækvivalenspunkt, mV og mL.



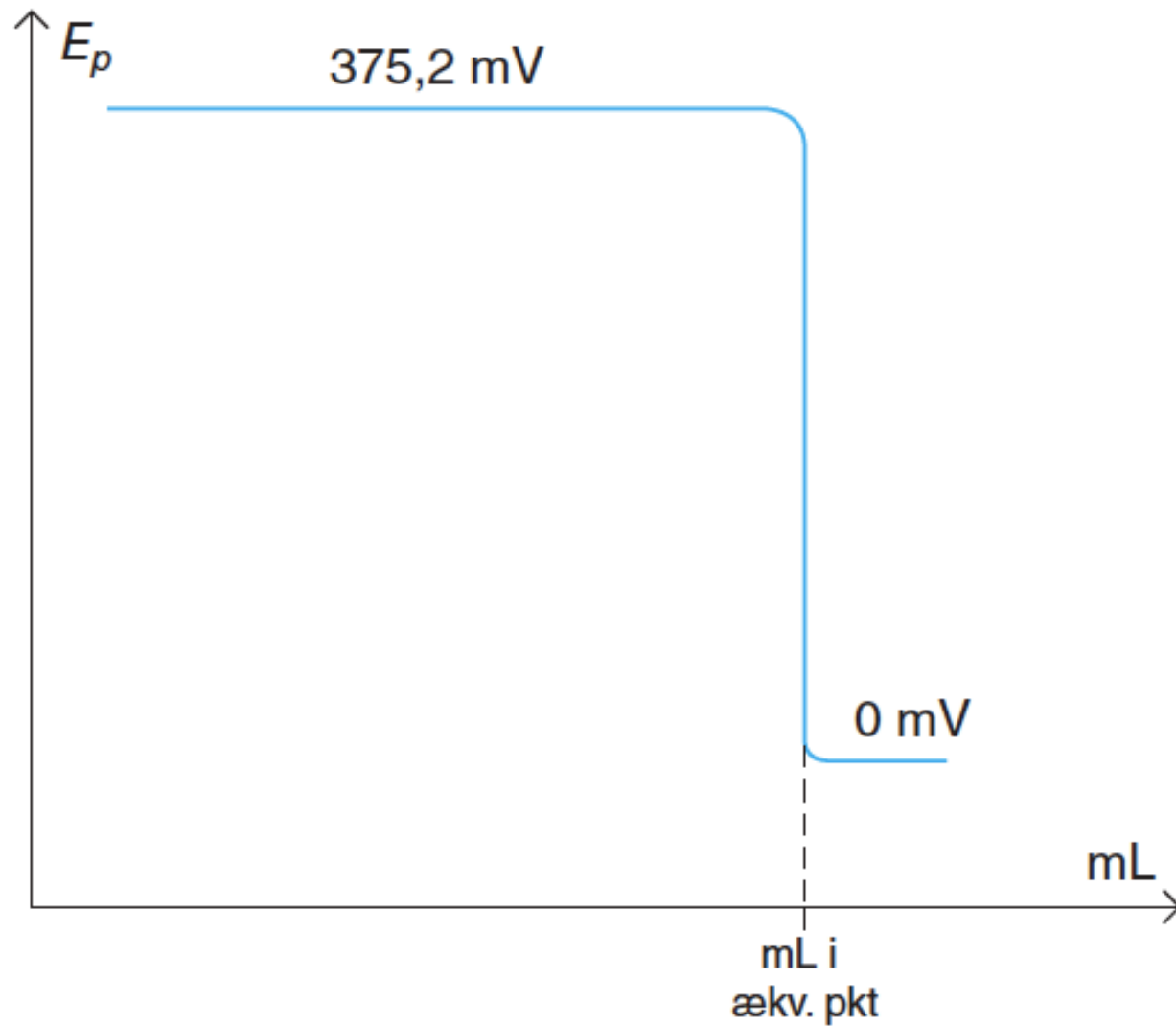
Figur 3.24. Pc-baseret autotitrators modulsammensætning.



Figur 3.25. Potentiometrisk titrercurve øverst og 1. afledede nederst.



Figur 3.26. Anvendelse af polarisationsadapter ved dead-stop end point-titrering.



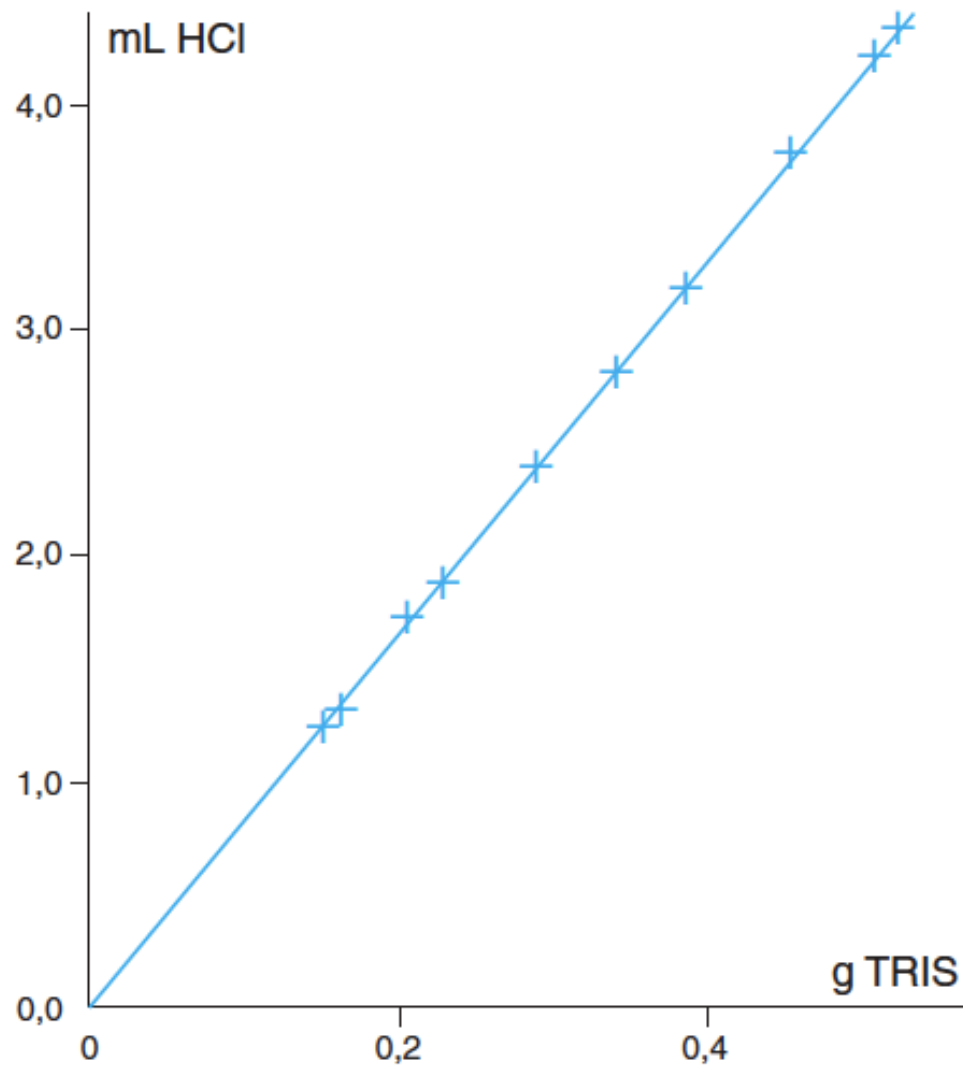
Figur 3.27. Titrerkurvens forløb ved redox-titrering.



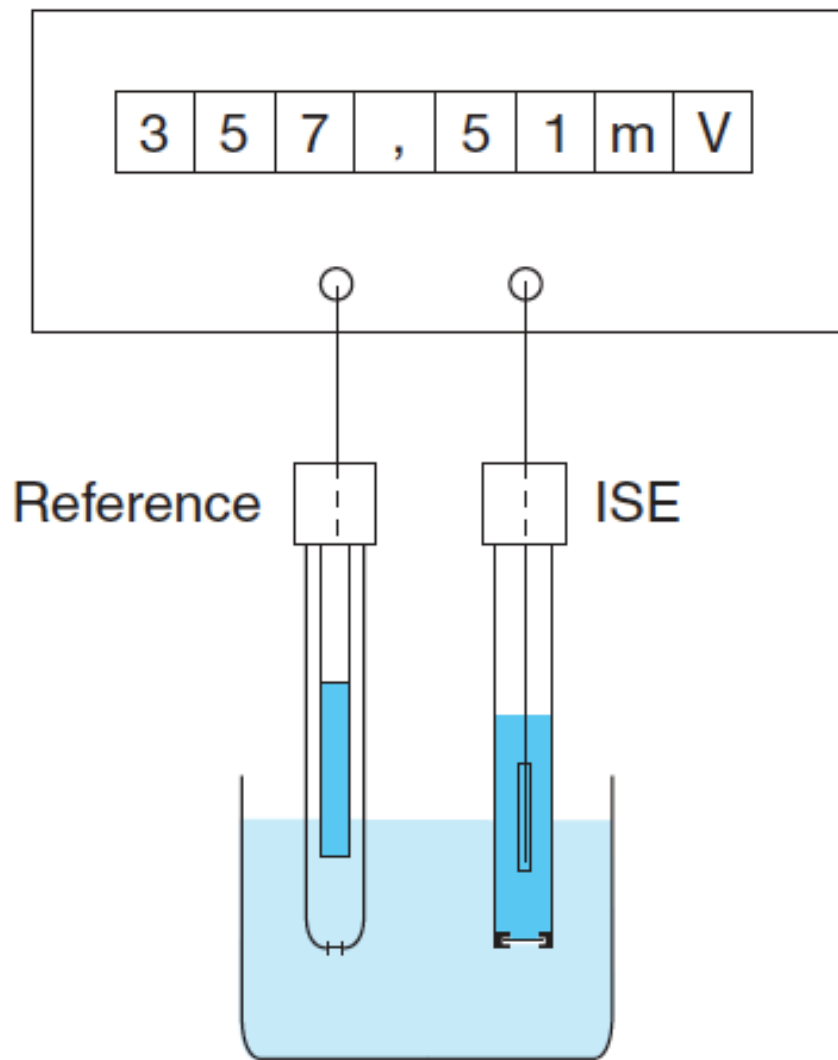
Figur 3.28. Volumetrisk Karl Fisher-titreringsudstyr.



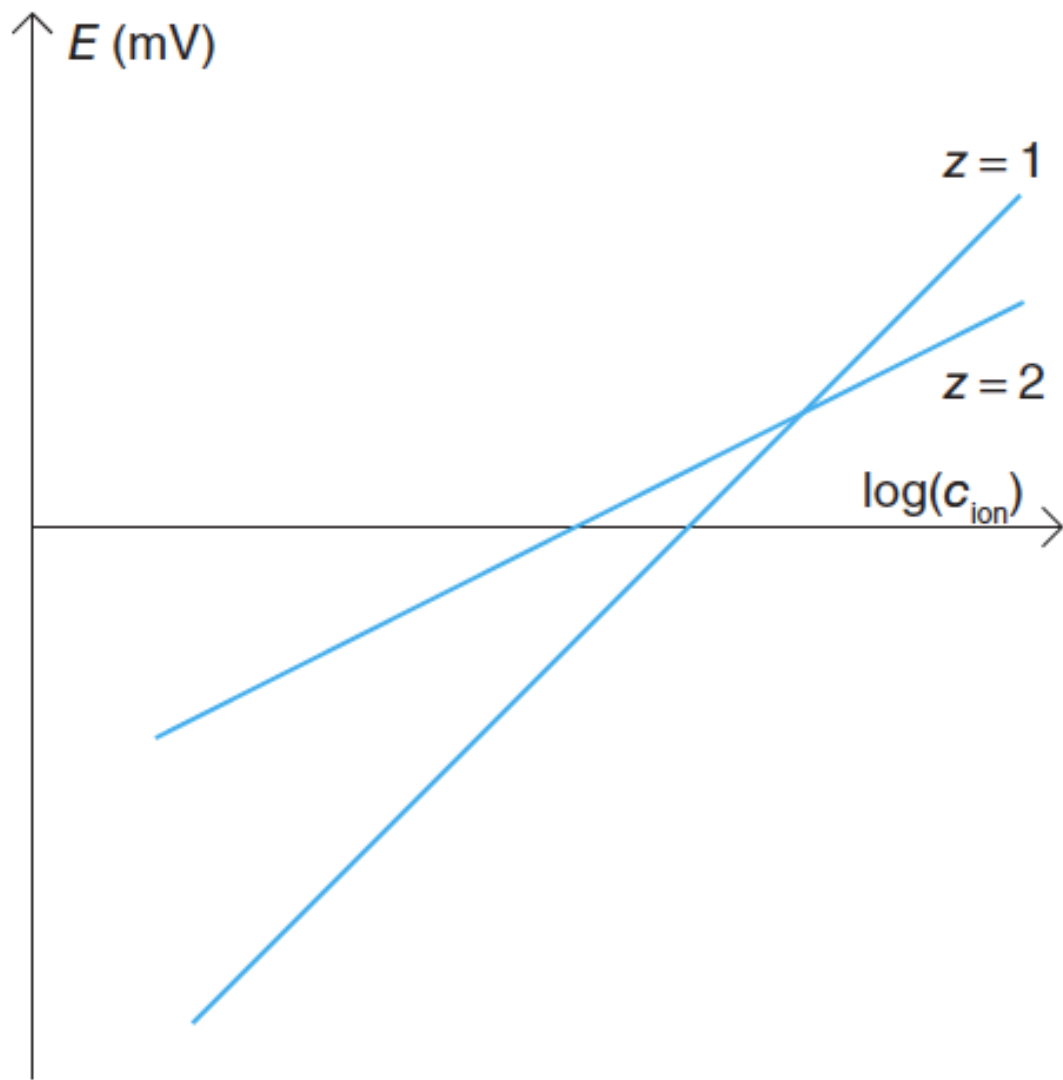
Figur 3.29. Columetrisk Karl Fisher-titreringsudstyr.



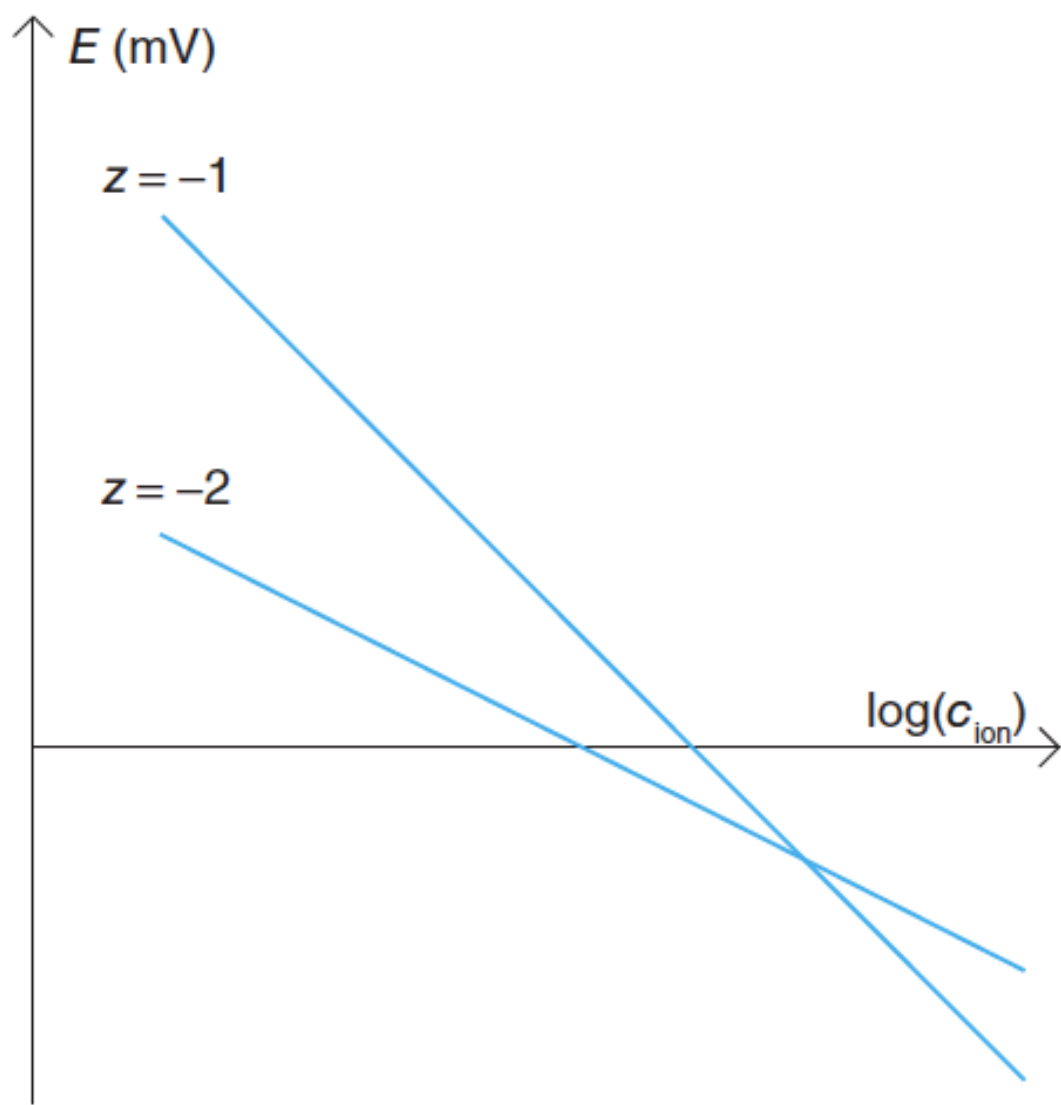
Figur 3.30. Graf til biasbestemmelse af mL HCl som funktion af g TRIS.



Figur 4.1. Opstilling til måling med ionselektiv elektrode.



Figur 4.2. Teoretisk sammenhæng mellem mV og $\log(c_{\text{ion}})$ for positive ioner.



Figur 4.3. Teoretisk sammenhæng mellem E mV og $\log(c_{\text{ion}})$ for negative ioner.

<i>Formel</i>	<i>Membran</i>	<i>Interferensjoner</i>
NH_4^+	PVC	K^+
Ca^{2+}	PVC	$\text{Zn}^{2+}, \text{Pb}^{2+}$
Cu^{2+}	Krystal	$\text{HgS}, \text{Ag}^+, \text{Cl}^-$
Pb^{2+}	Krystal	$\text{Hg}^{2+}, \text{Cu}^{2+}, \text{Ag}^+$
K^+	PVC	$\text{NH}_4^+, \text{Na}^+$
Na^+	Glas	$\text{Li}^+, \text{NH}_4^+, \text{Ag}^+$
Ag^+	Krystal	Hg^{2+}
Br^-	Krystal	$\text{I}^-, \text{SCN}^-, \text{CN}^-, \text{S}^{2-}$
Cl^-	Krystal	$\text{I}^-, \text{SCN}^-, \text{CN}^-, \text{Br}^-$
F^-	Krystal	OH^-
I^-	Krystal	$\text{Br}^-, \text{SCN}^-, \text{CN}^-, \text{Hg}^{2-}$
NO_3^-	PVC	$\text{Cl}^-, \text{Br}^-, \text{NO}_2^-$
S^{2-}	Krystal	Hg^{2+}

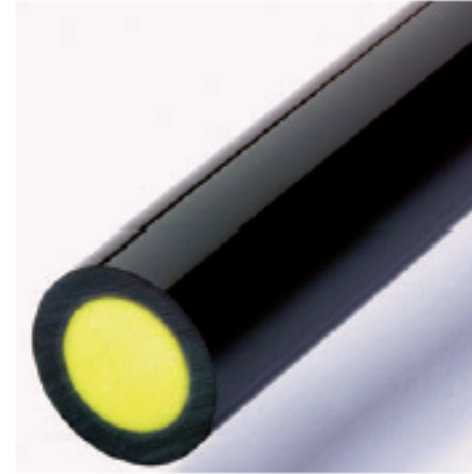
Tabel 4.1. ISE



Calcium, PVC

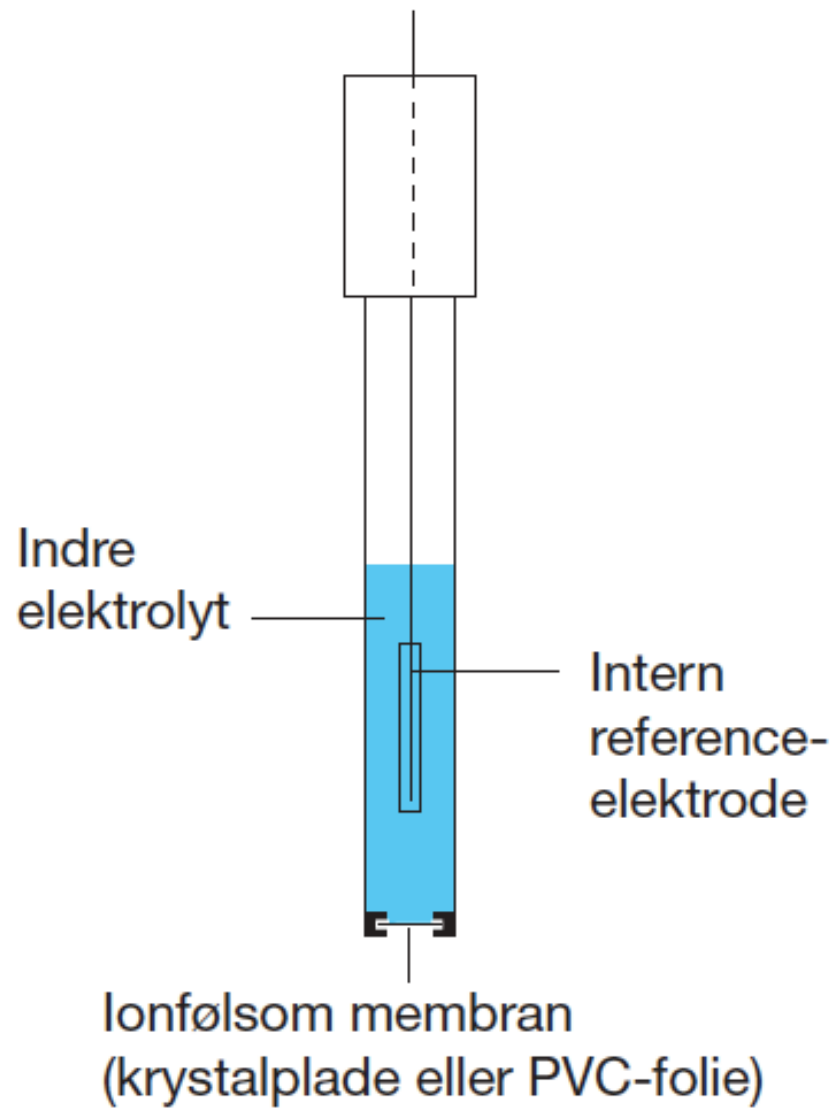


Natrium, Glas

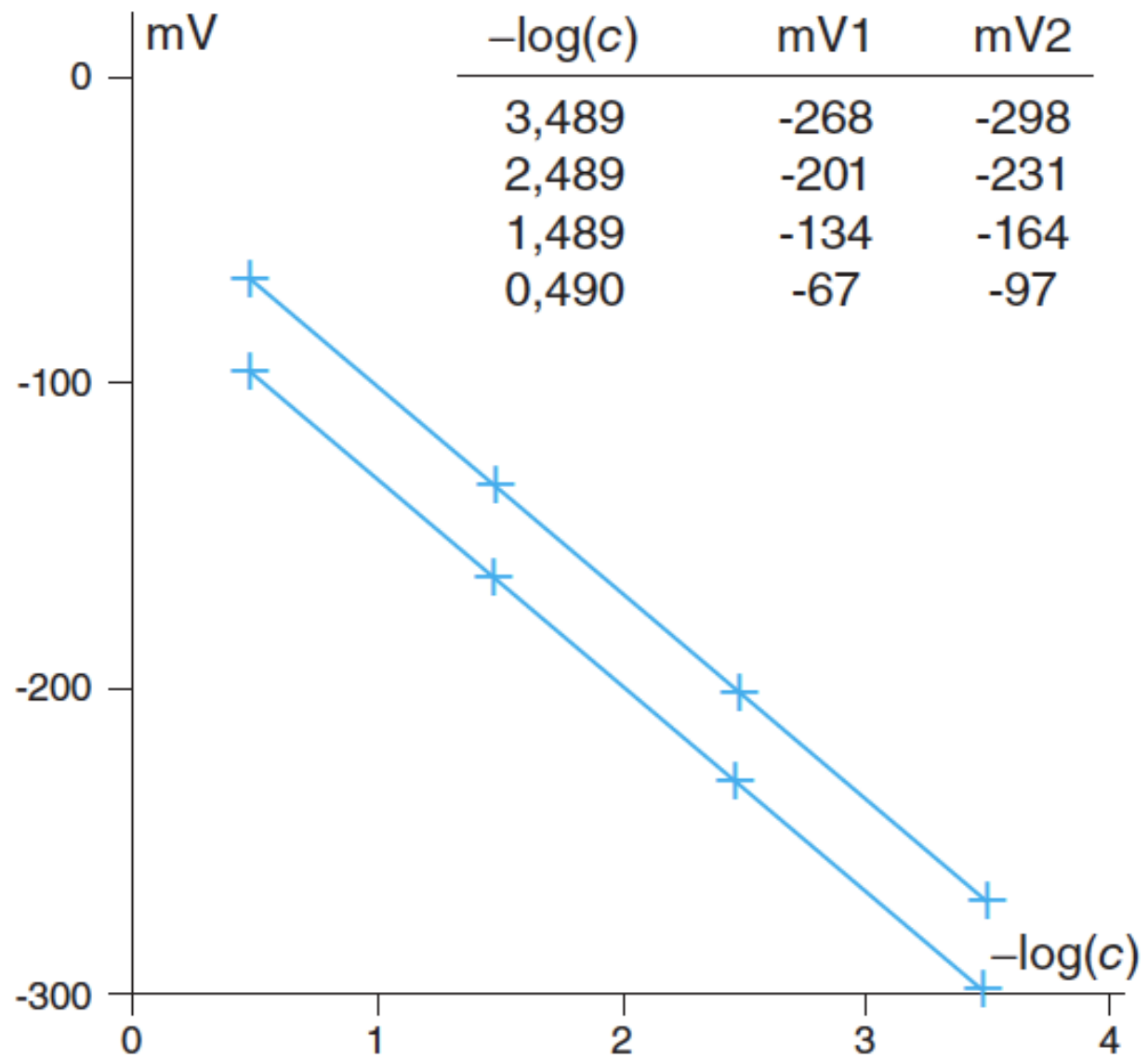


Cyanid, Krystal

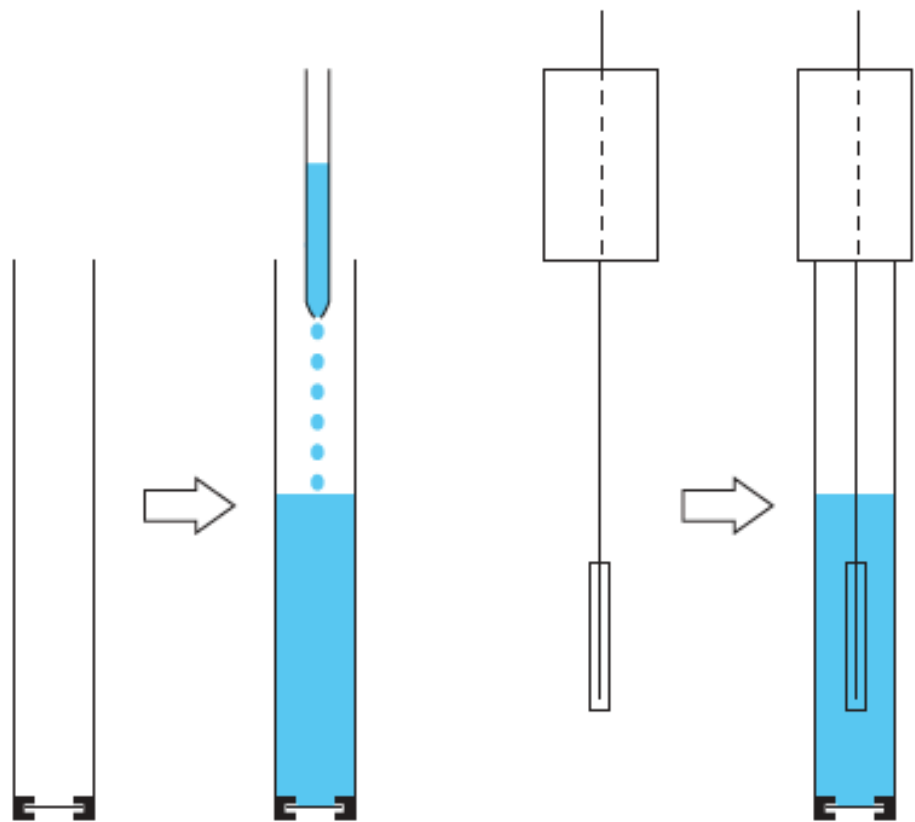
Figur 4.4. Tre membrantyper på elektroder.



Figur 4.5. Principiel opbygning af en ionselektiv elektrode.



Figur 4.6. Ionselektiv elektrodes potentialedrft.



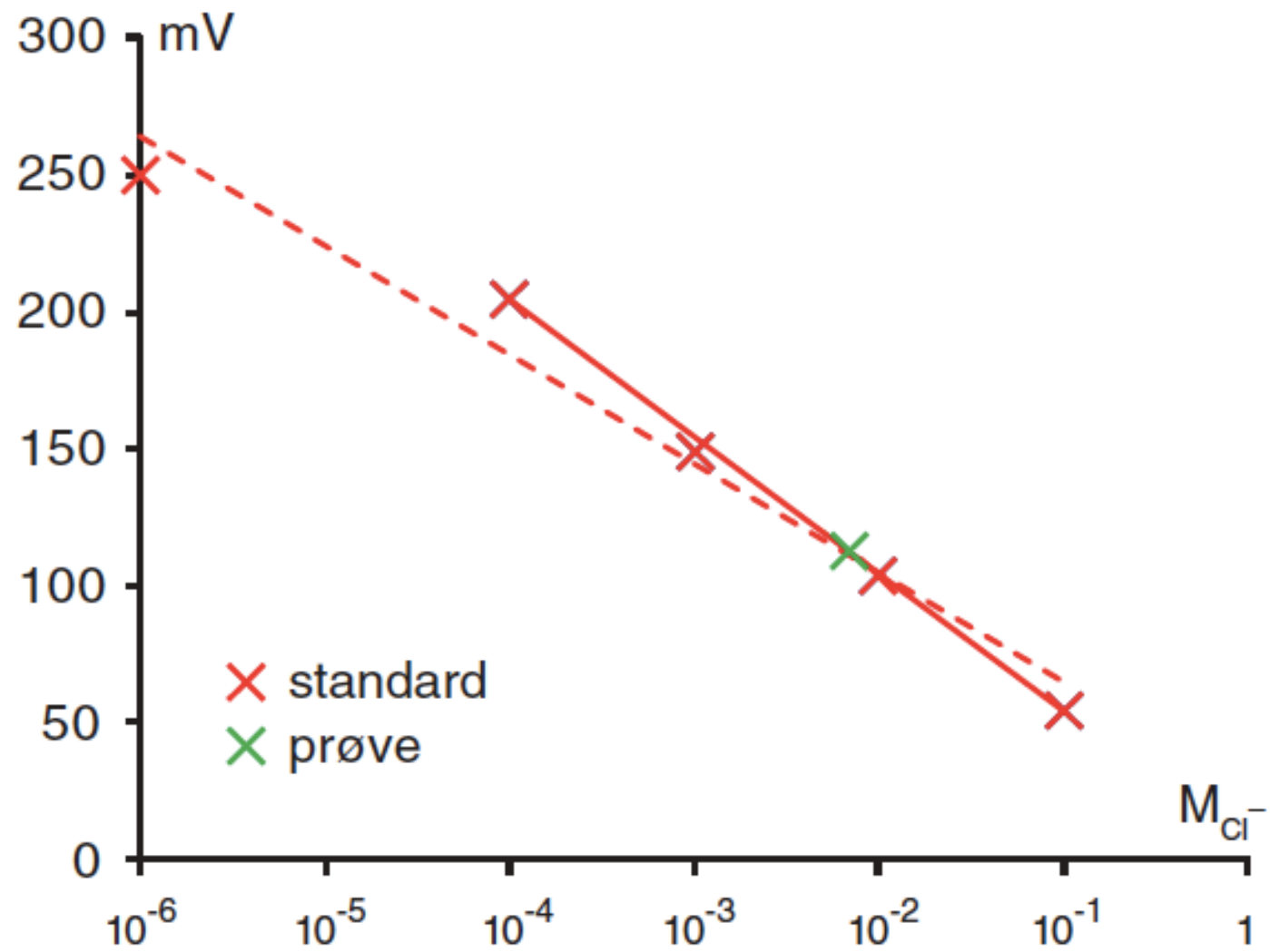
Udskifteligt
elektrode-
kammer
med
membran
i bunden

Elektrode-
kammer
påfyldes
elektrolyt

Indre
reference-
elektrode
(AgCl | Ag)

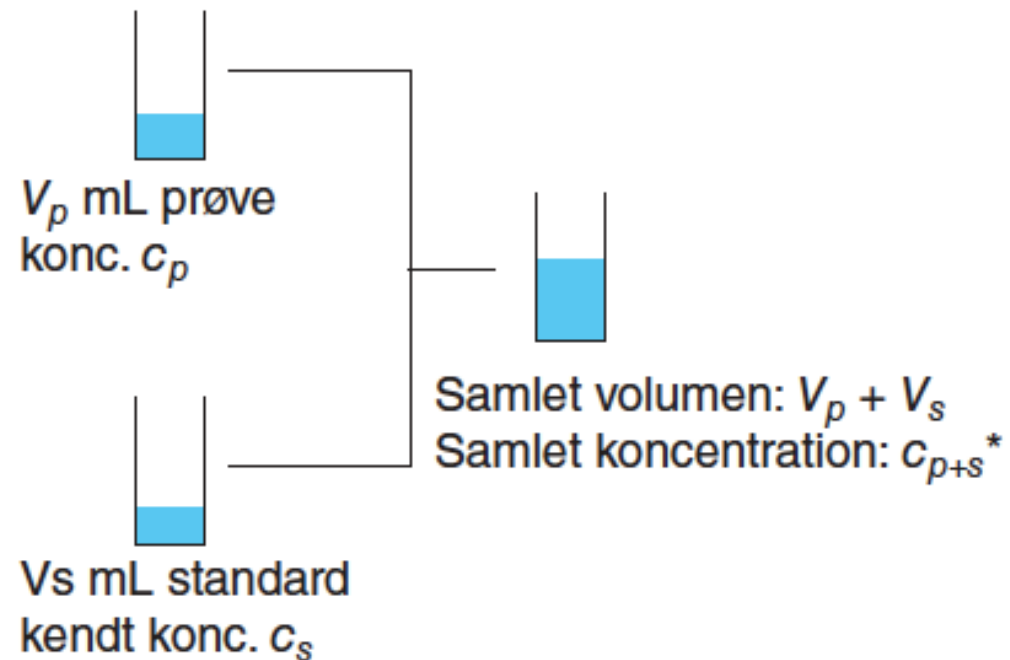
Elektrode-
kammer
og indre
reference-
elektrode
er samlet

Figur 4.7. Samling af ISE med udskiftelig membran.



Figur 4.8. Kalibreringskurve til chloridbestemmelse ved direkte potentiometri.

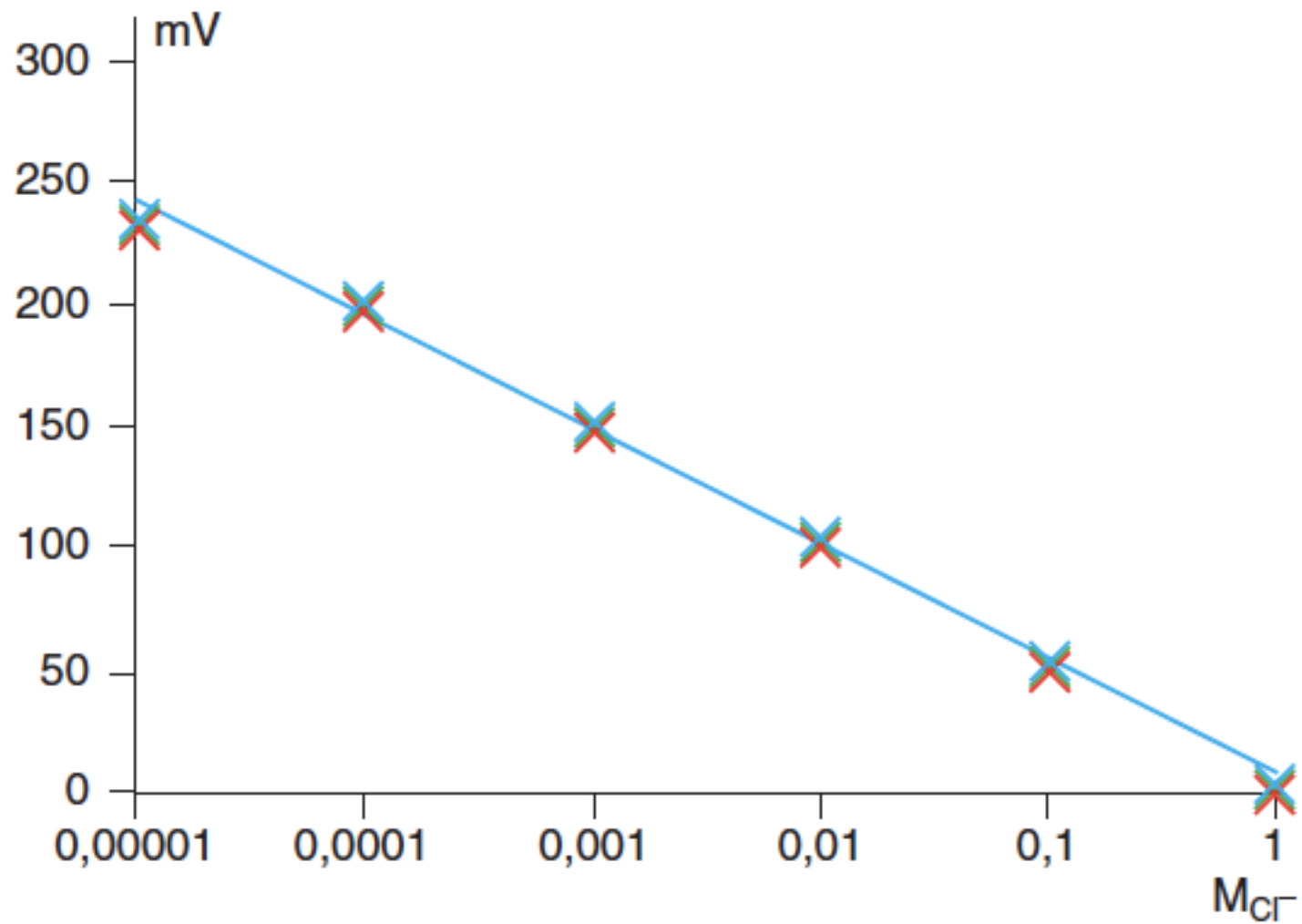
Standardaddition



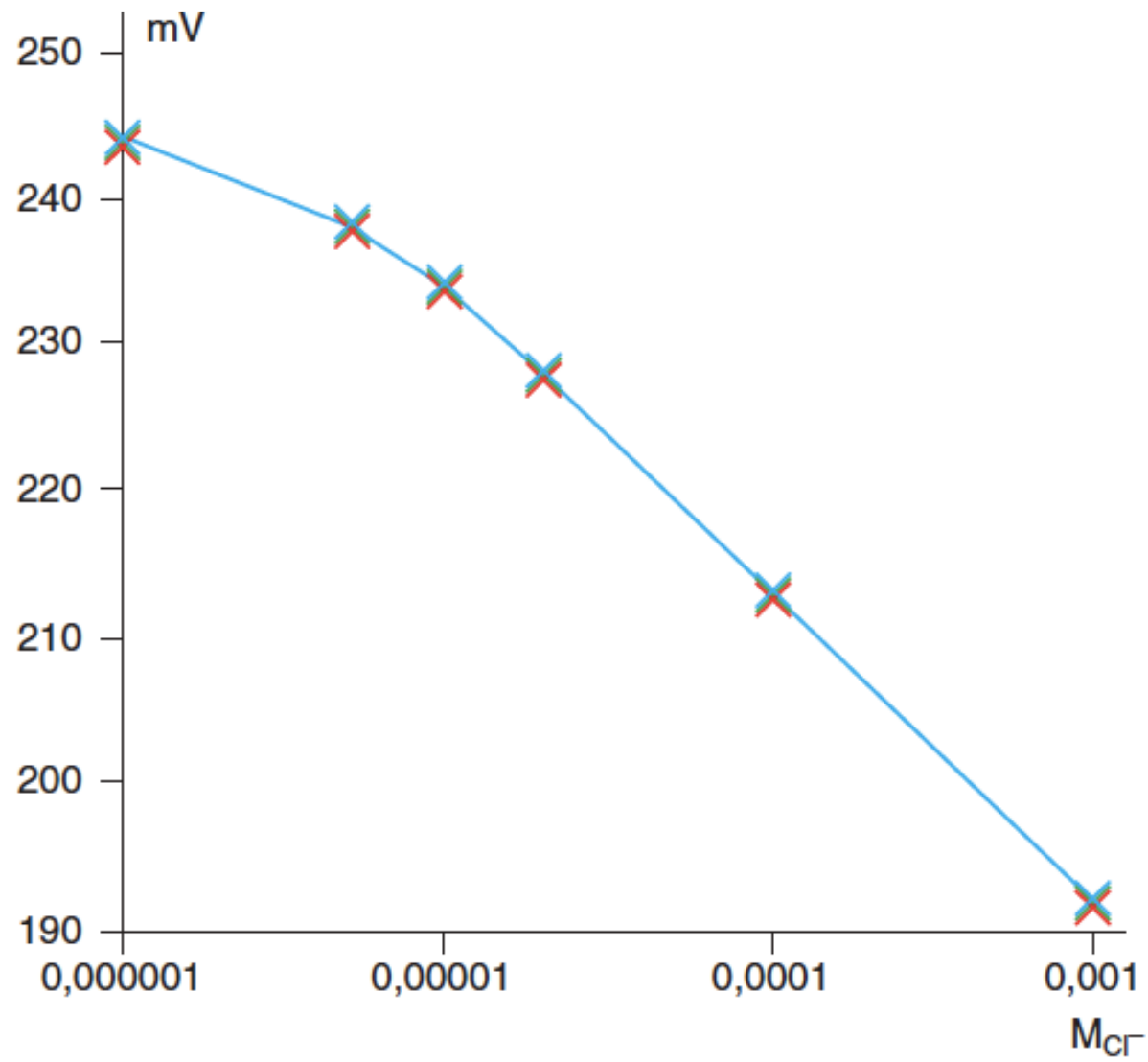
$$*c_{p+s} = c_p \cdot \frac{V_p}{V_p + V_s} + c_s \cdot \frac{V_s}{V_p + V_s}$$

$$c_{p+s} = \frac{c_p V_p + c_s V_s}{V_p + V_s}$$

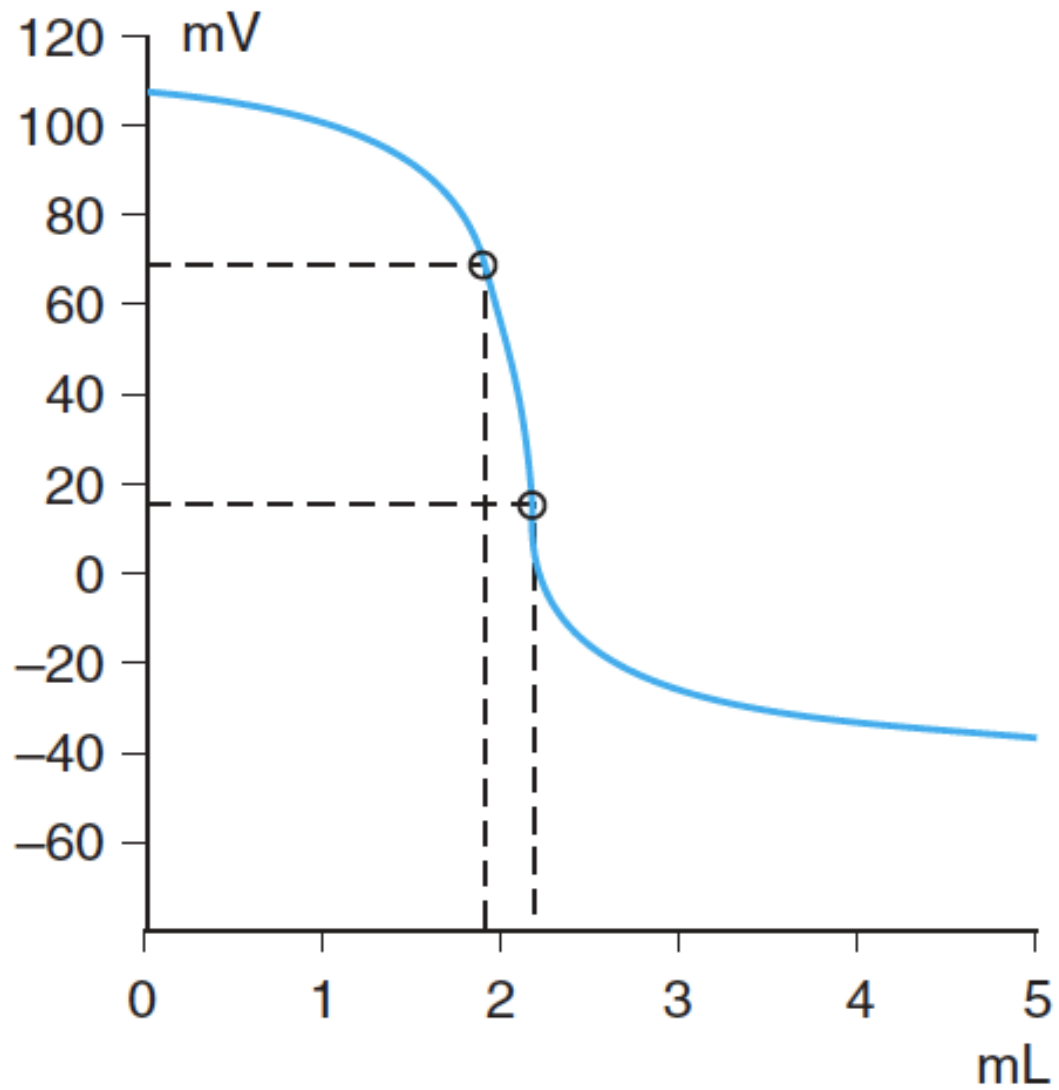
Figur 4.9. Standardaddition og beregning af blandingens koncentration.



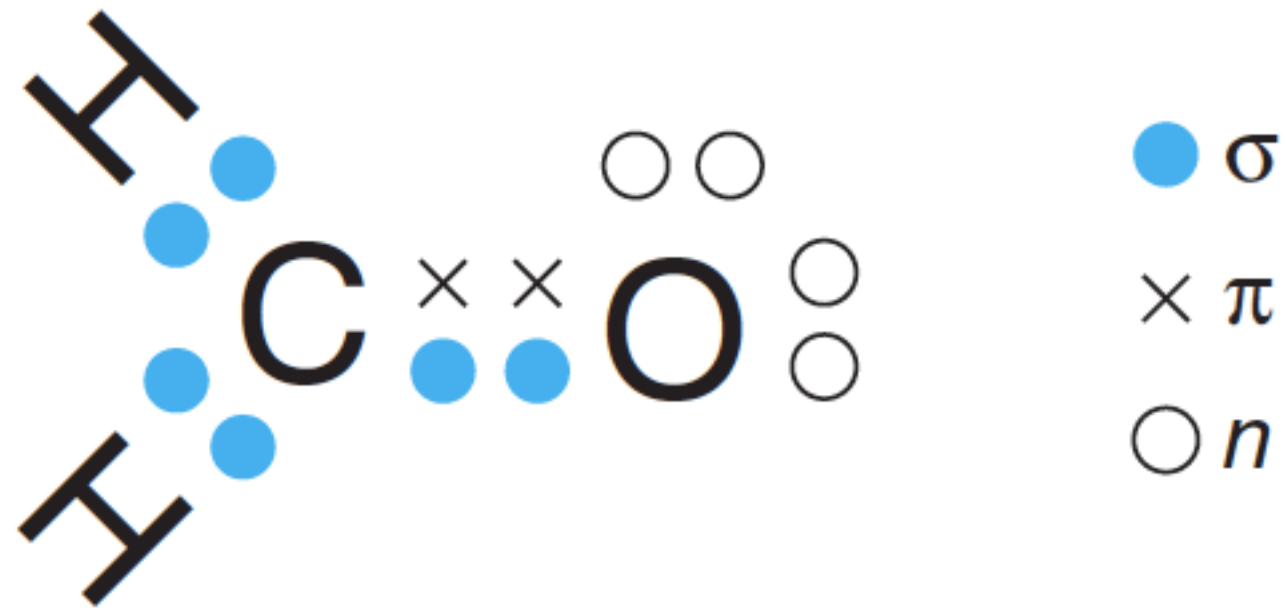
Figur 4.10. Linearitetsbestemmelse med chlorid-ISE



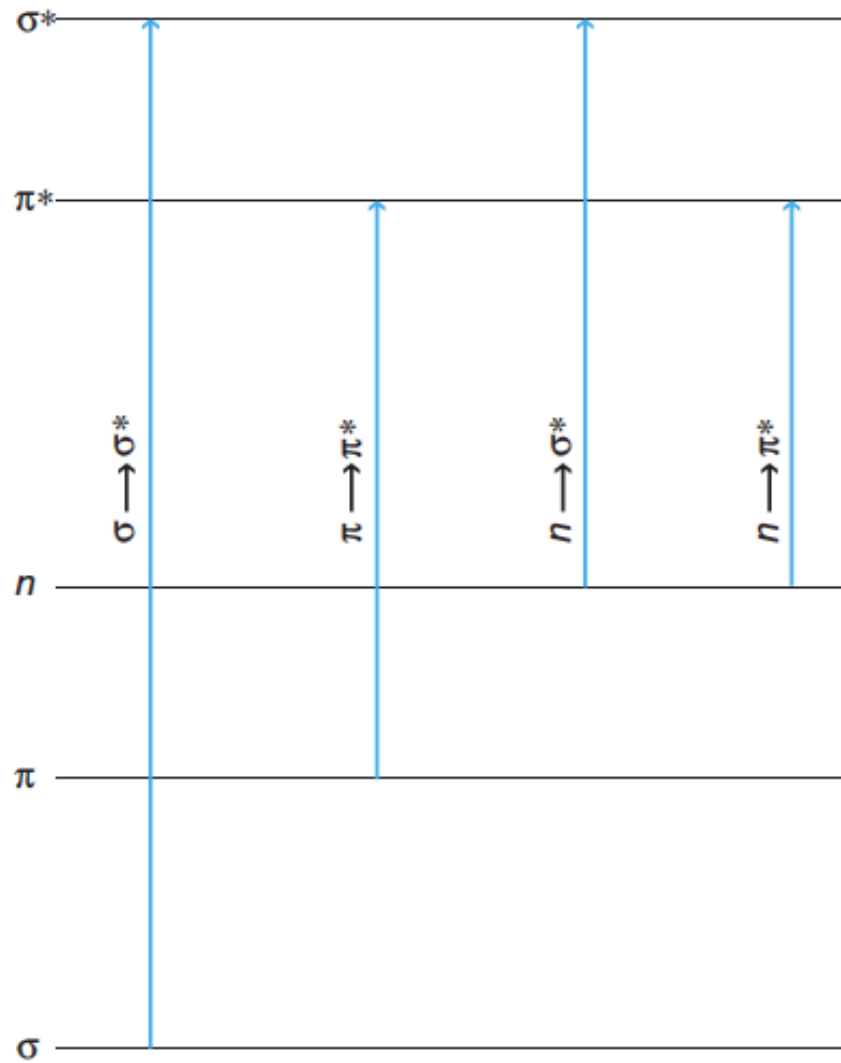
Figur 4.11. Bestemmelse af kvantifikationsgrænse for chlorid-ISE



Figur 4.12. Titrerkurve af Ca²⁺ og Mg²⁺ i vand med EDTA.



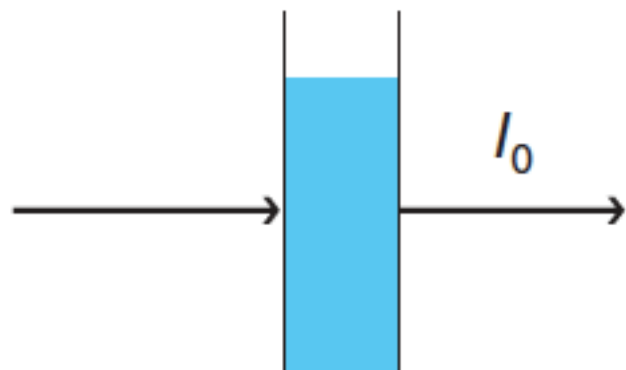
Figur 5.1. σ , π og n -elektroner.



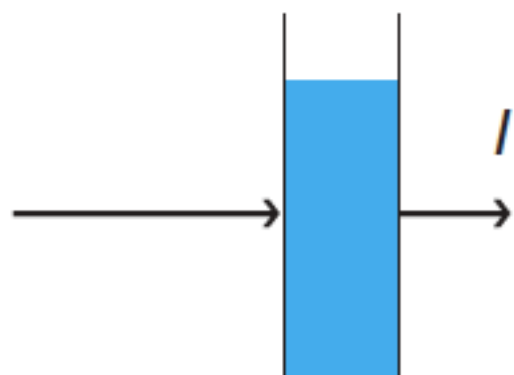
Figur 5.2. Nogle af elektronernes energispring i et molekyle.

<i>Chromofor</i>	<i>Gruppe/stof</i>	$\lambda_{max} nm$
Aldehyd	-CHO	210
Amin	-NH ₂	195
Benzen	C ₆ H ₆	184, 202, 255
Carboxyl	-COOH	200-210
Ester	-COOR	205
Ether	-O-	185
Ethylen	-C=C-	190
Iodid	-I	260
Keton	>C=O	195
MnO ₄ ⁻		525
Nitrat	-ONO ₂	270
Nitrit	-ONO	220-230
Phenylalanin		257
Toluen	C ₆ H ₅ CH ₃	207, 261
Tryptophan		280, 219

Tabel 5.1. Chromoforer.



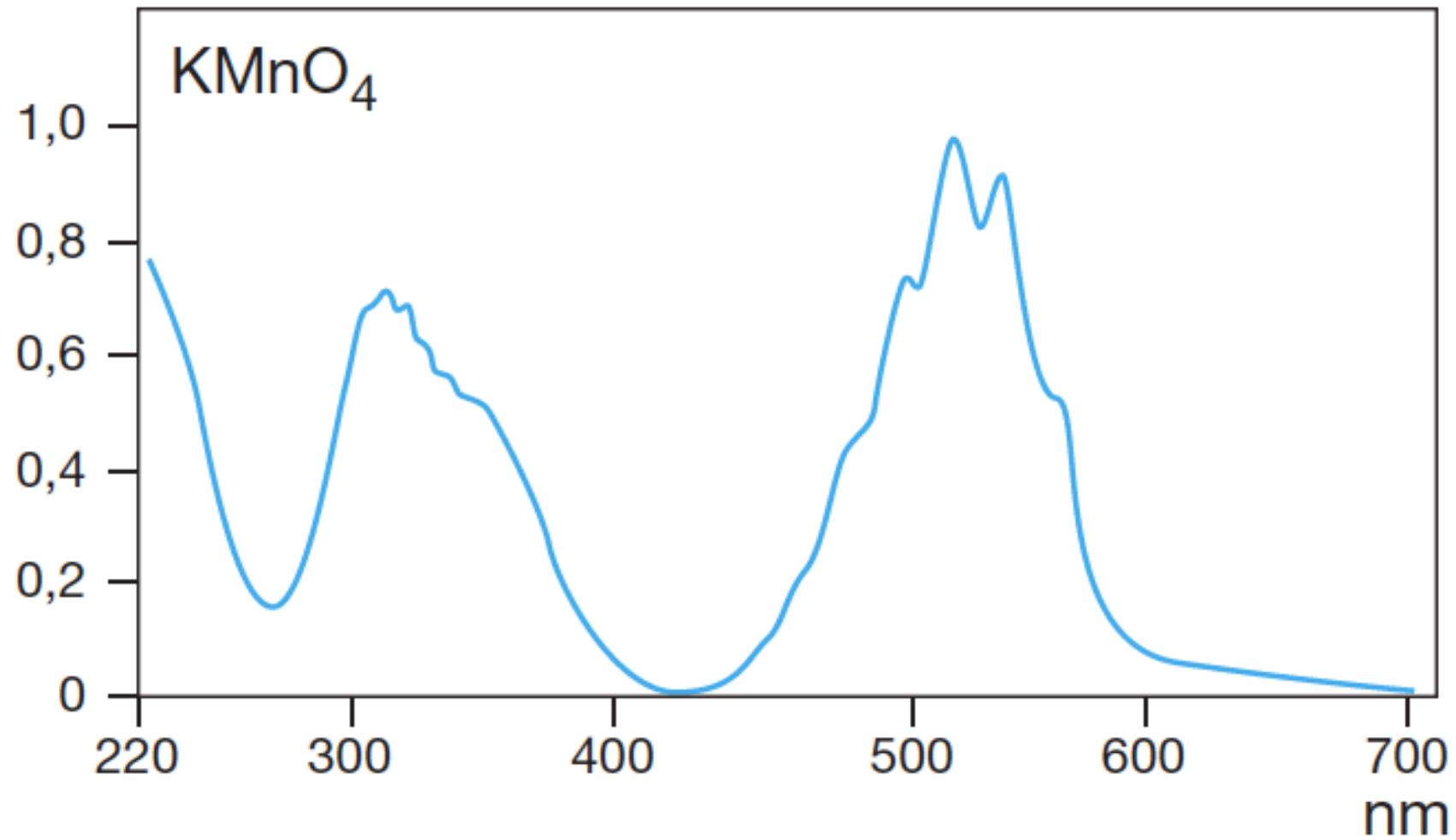
Blindprøve



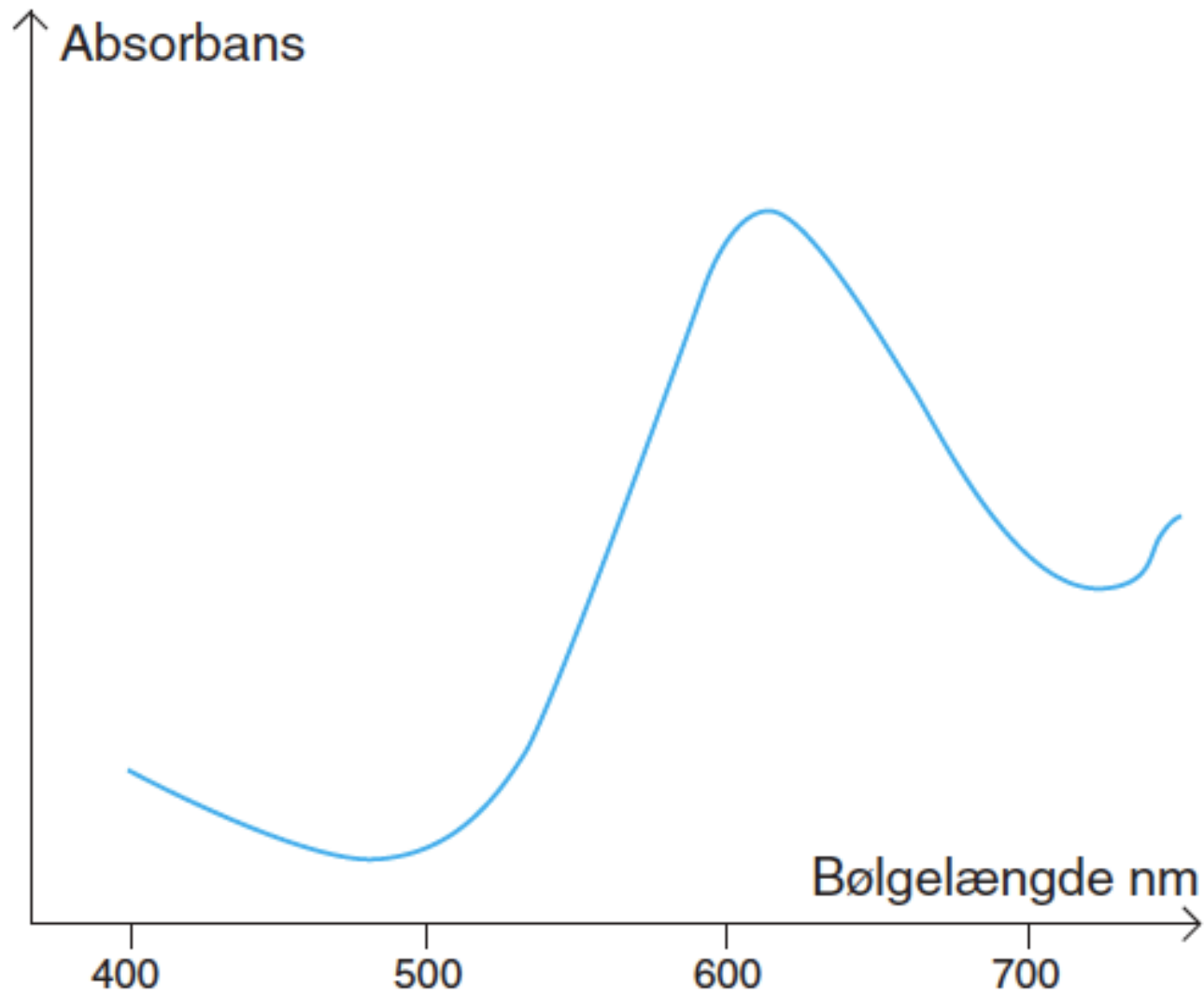
Prøve med analyt

Figur 5.3. Måling i forhold til blindprøve.

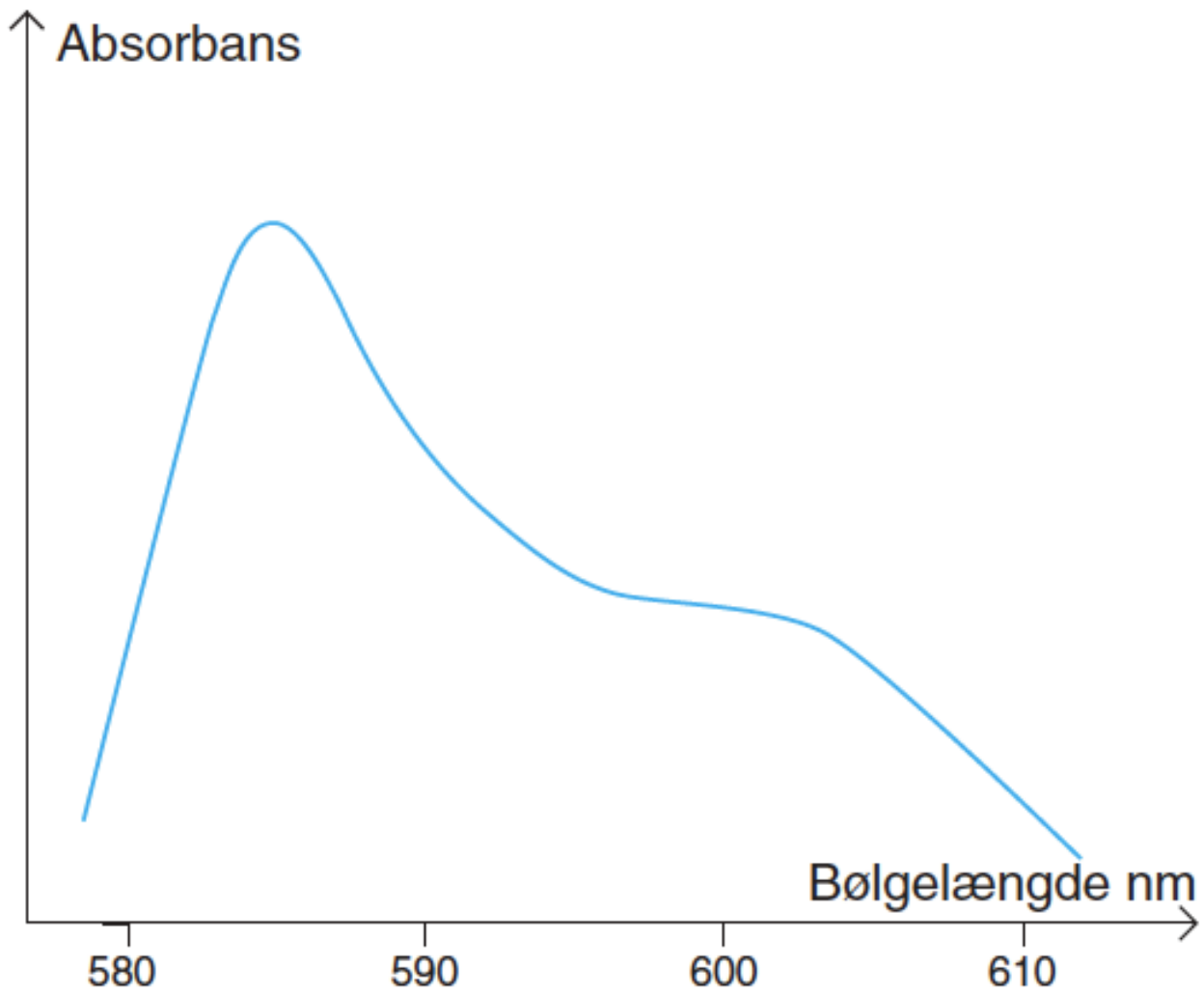
Absorbans



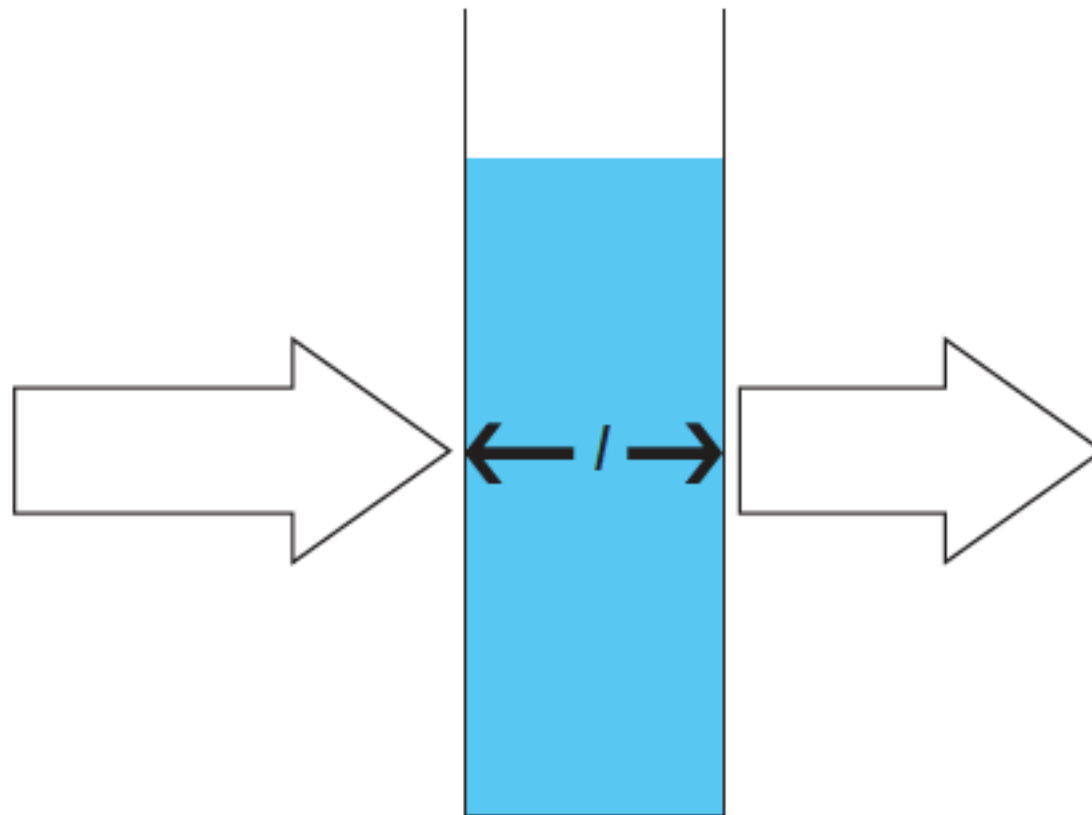
Figur 5.4. Absorptionsspektrum.



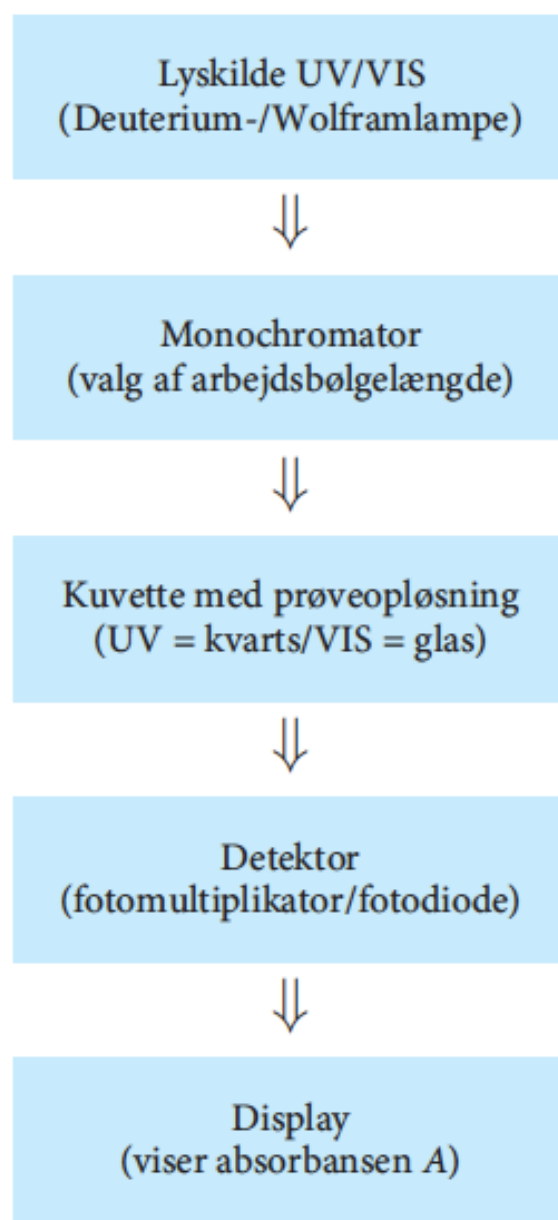
Figur 5.5. Bestemmelse af arbejdsbølgelængde.



Figur 5.6. Arbejdsbølgelængde, måling i snævert bølgelængdeinterval.



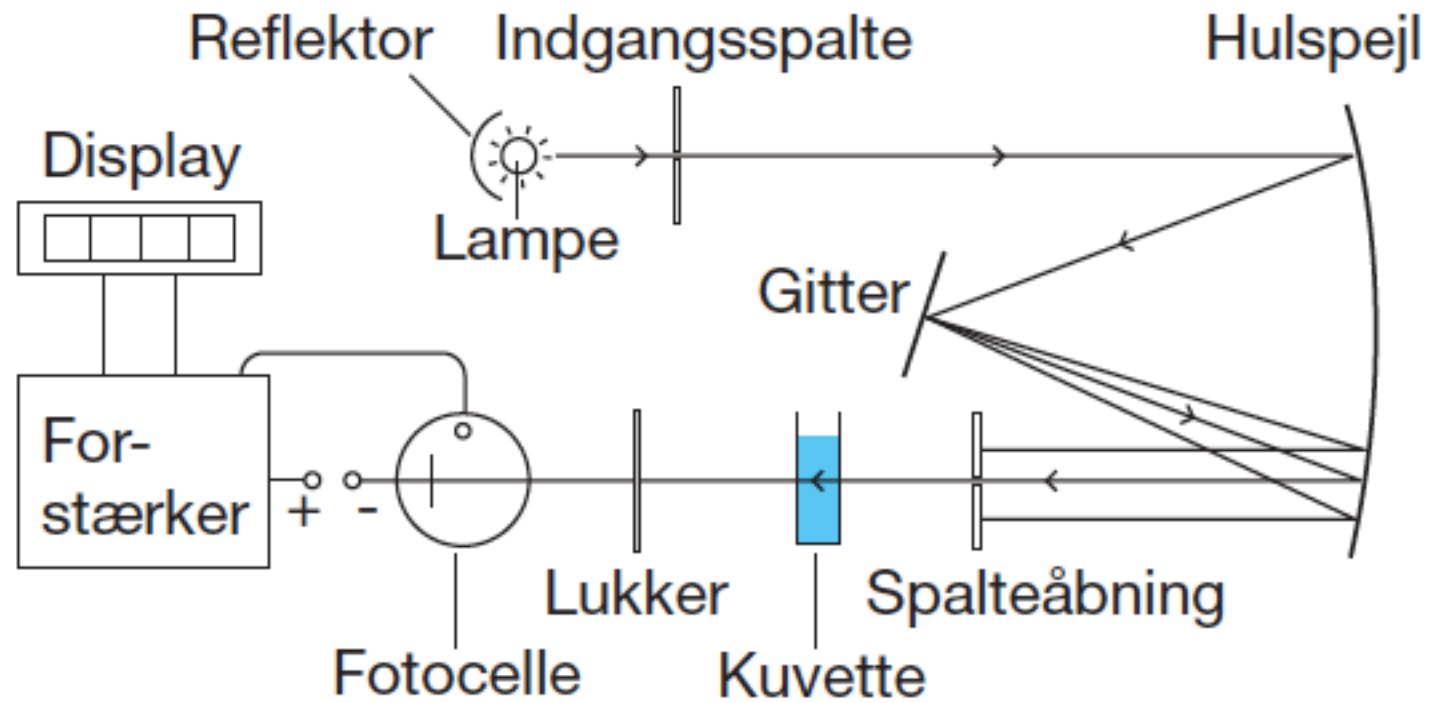
Figur 5.7. Lysvejen i Lambert-Beers lov.



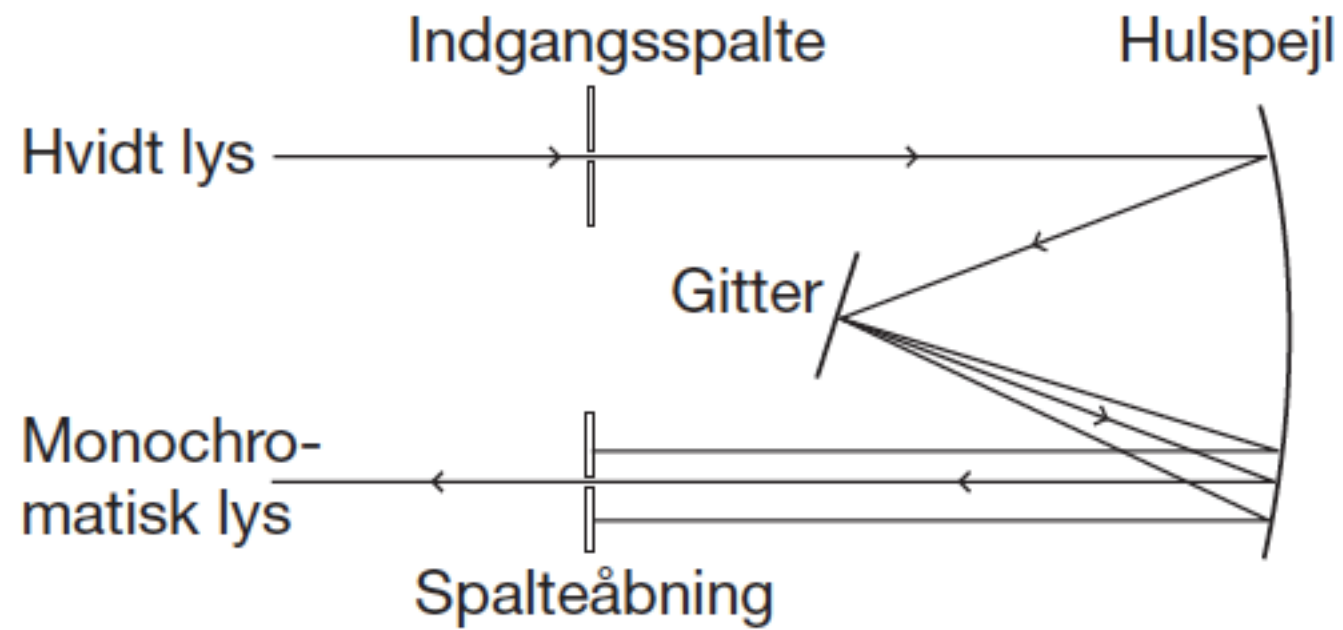
Figur 5.8. Spektrofotometrets opbygning.



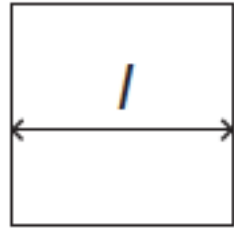
Figur 5.9. Spektrofotometer.



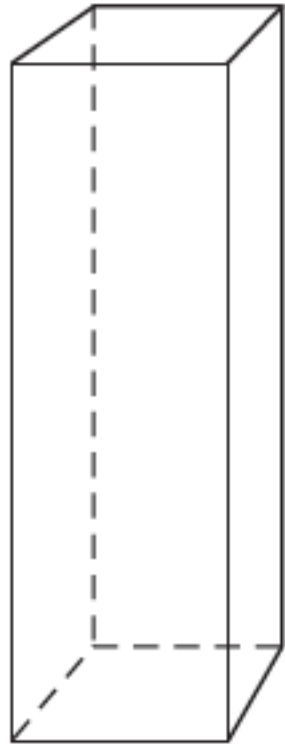
Figur 5.10. Spektrofotometrets opbygning i detaljer.



Figur 5.11. Monochromatorens opbygning.



Set ovenfra

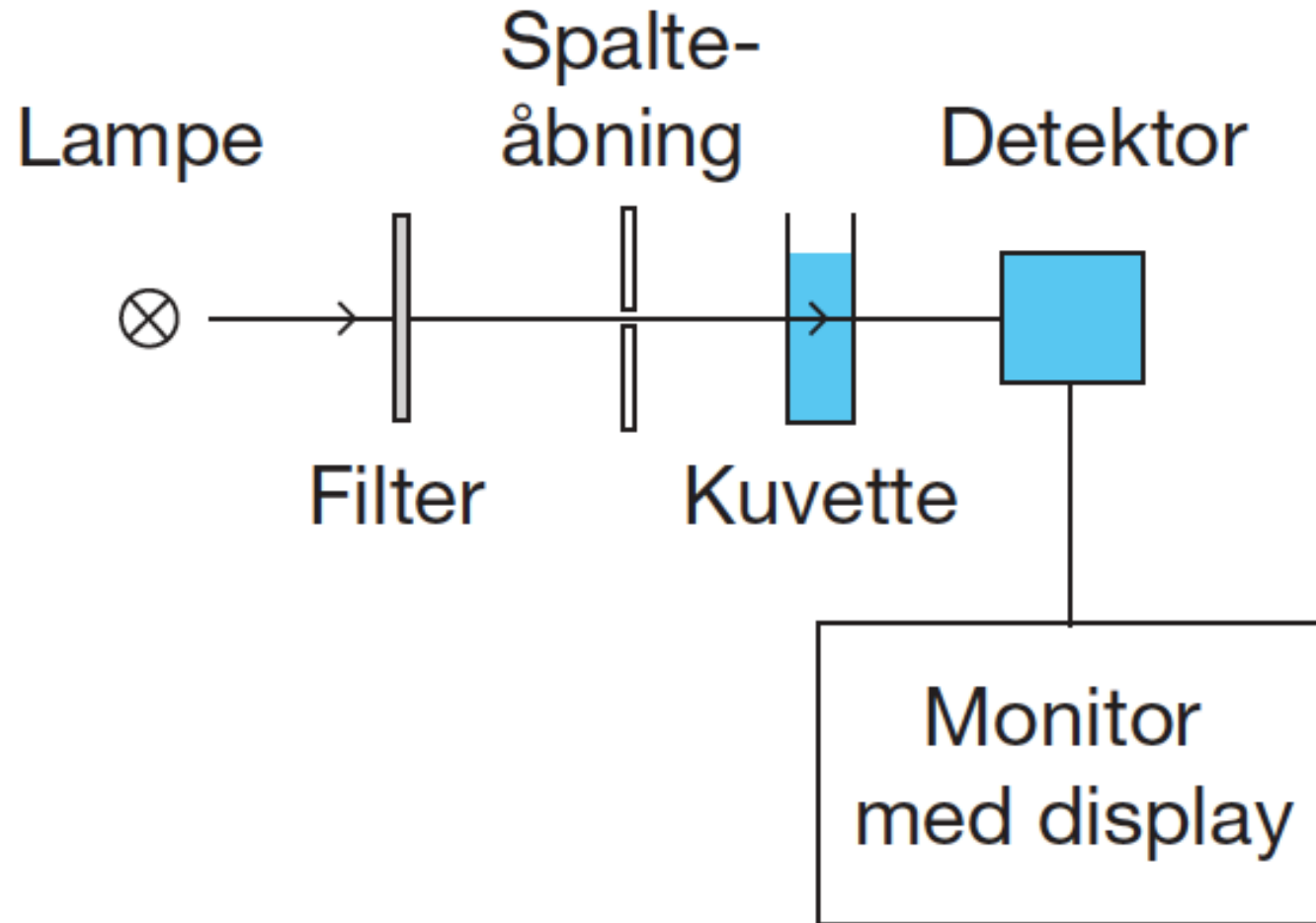


Set fra siden

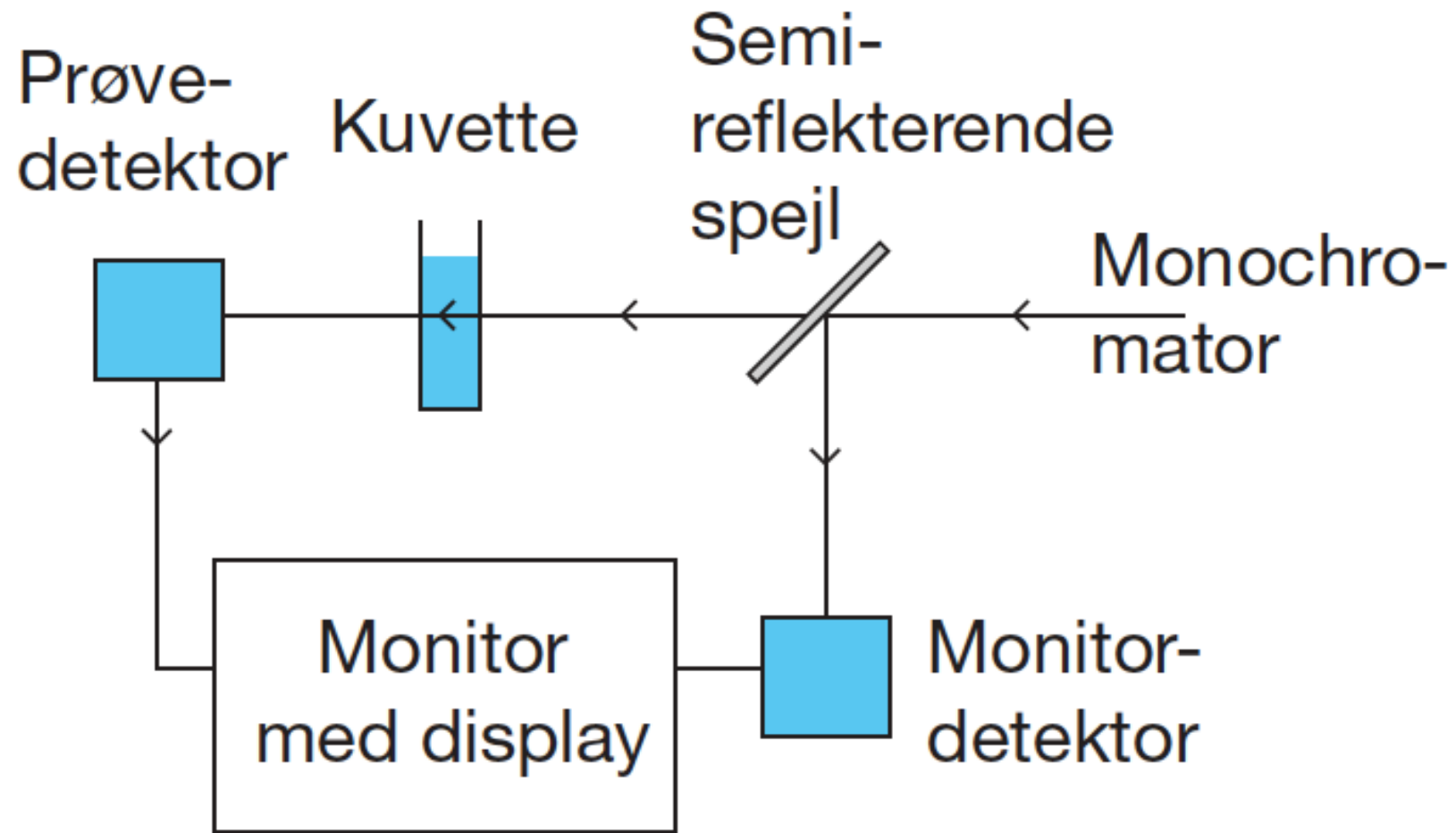
Figur 5.12. Standardkuvette.



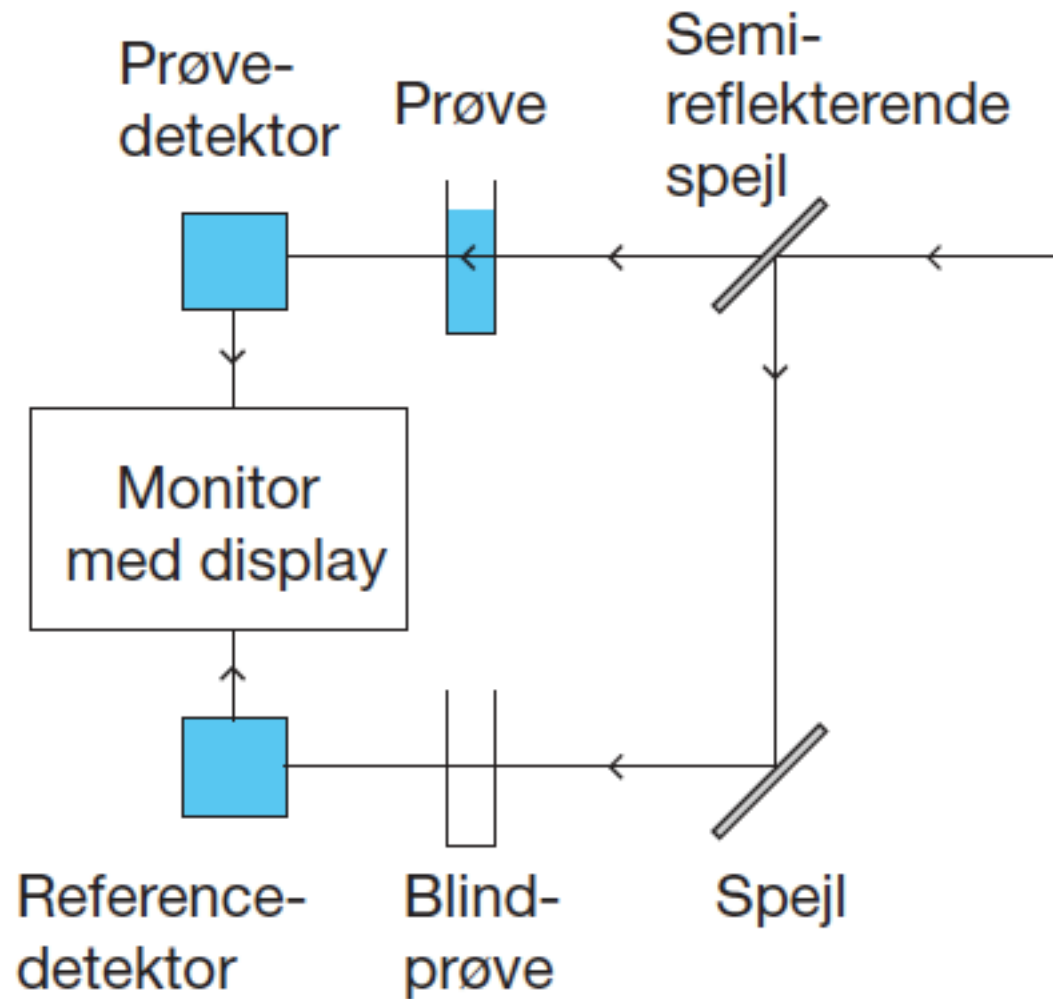
Figur 5.13. Nanokuvette⁶⁷.



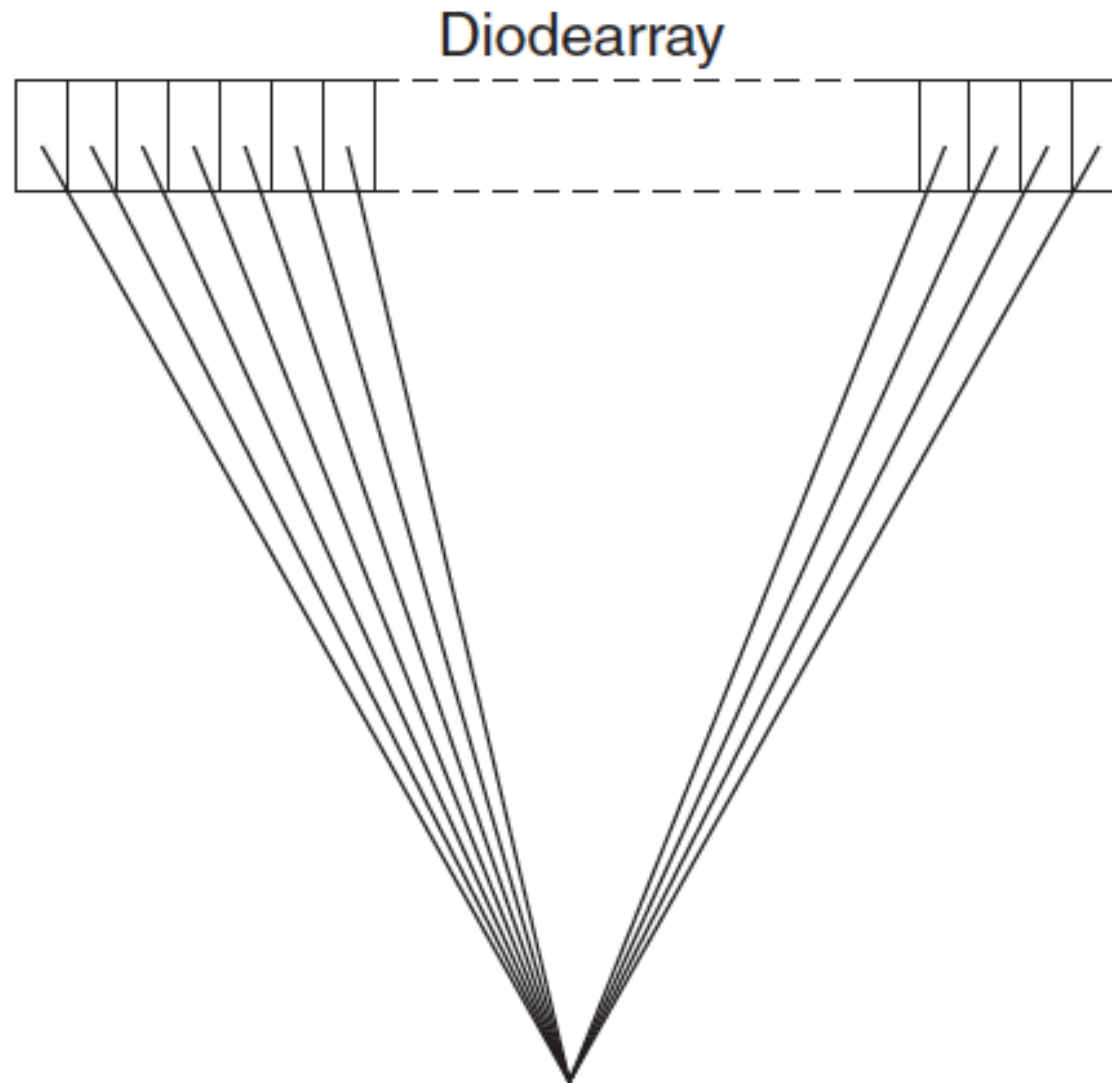
Figur 5.14. Filterfotometrets opbygning.



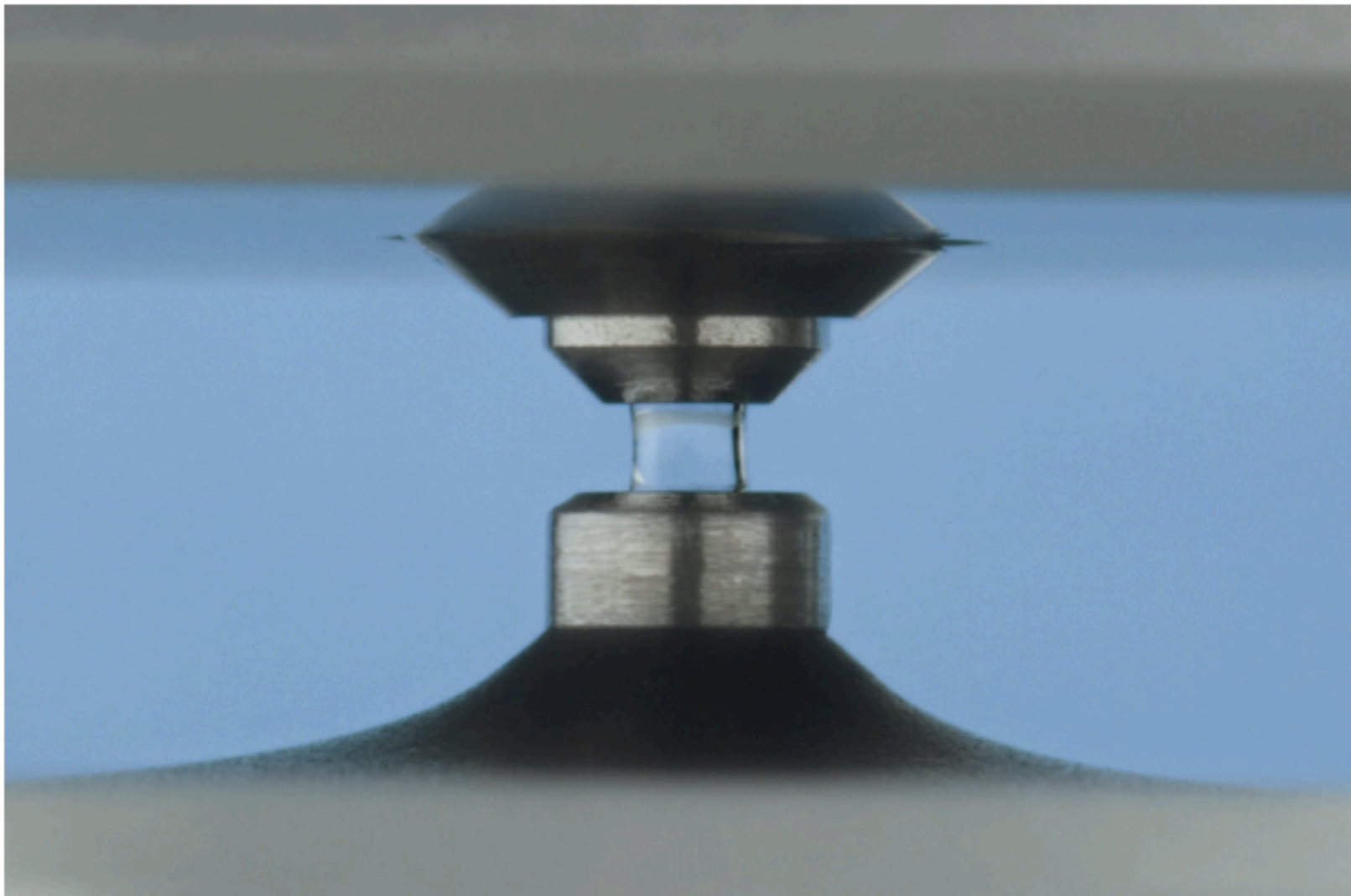
Figur 5.15. Enkeltstråle-spektrofotometer.



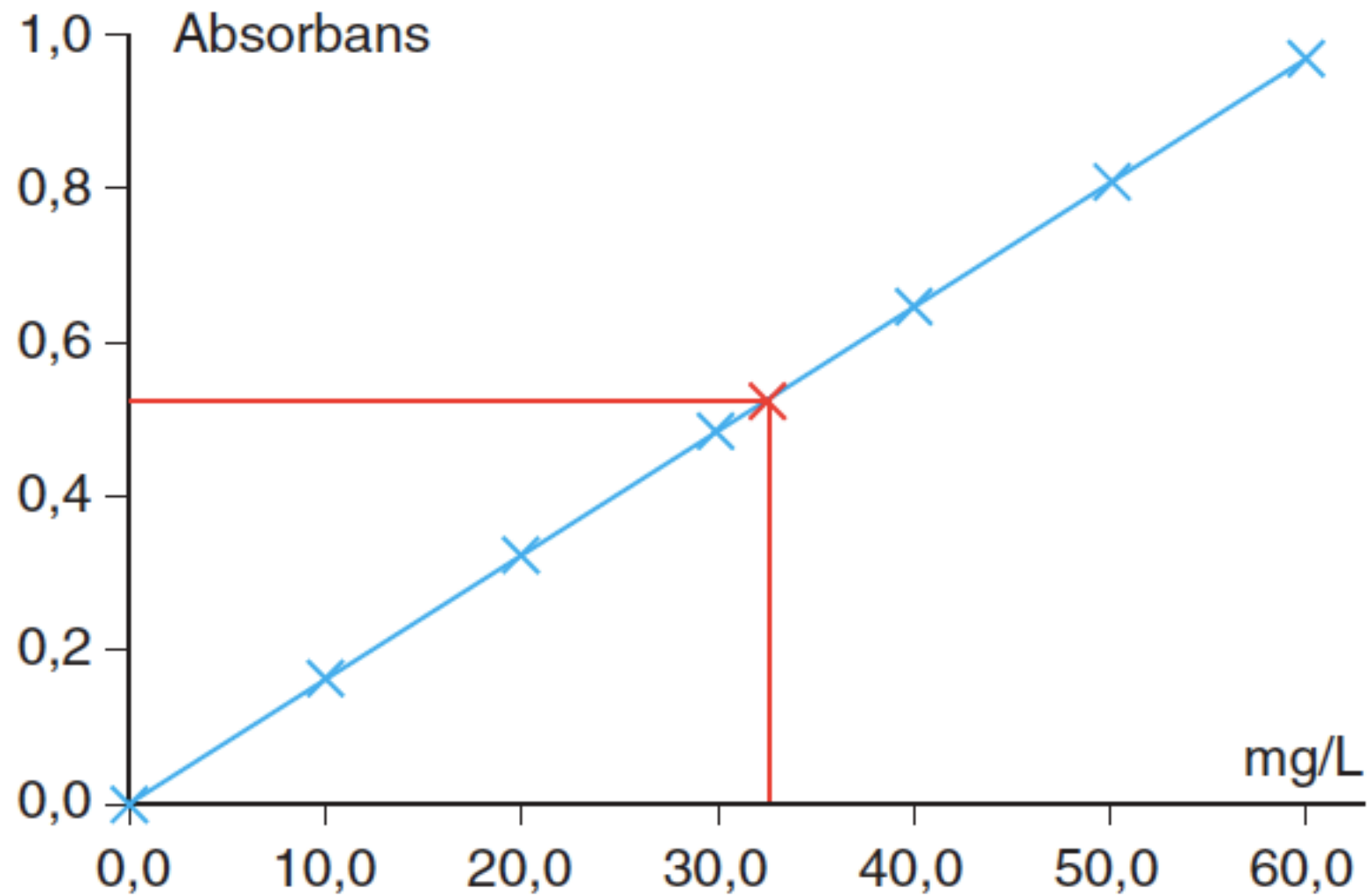
Figur 5.16. Dobbeltstråle-spektrofotometer.



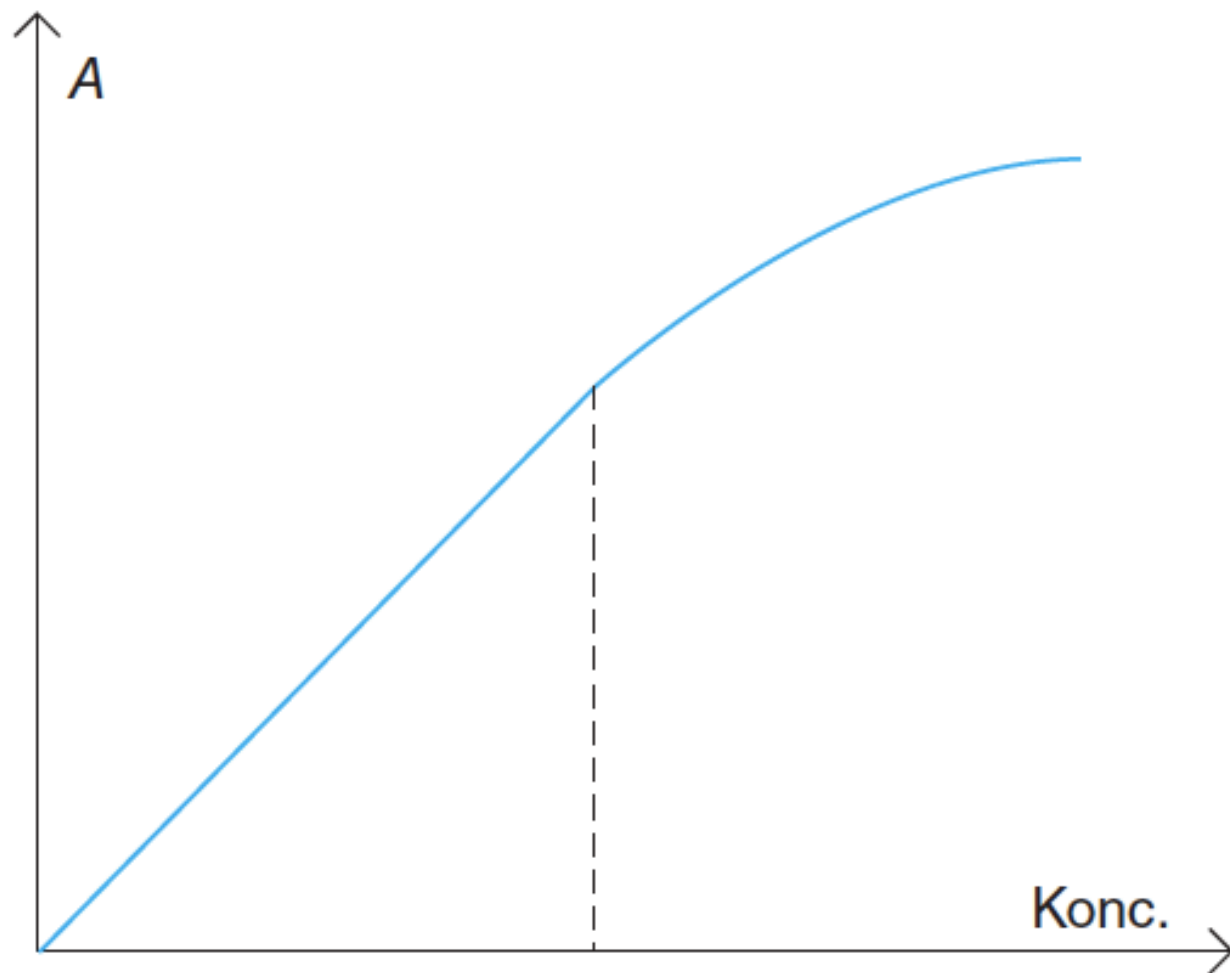
Figur 5.17. Diodearraydetektor.



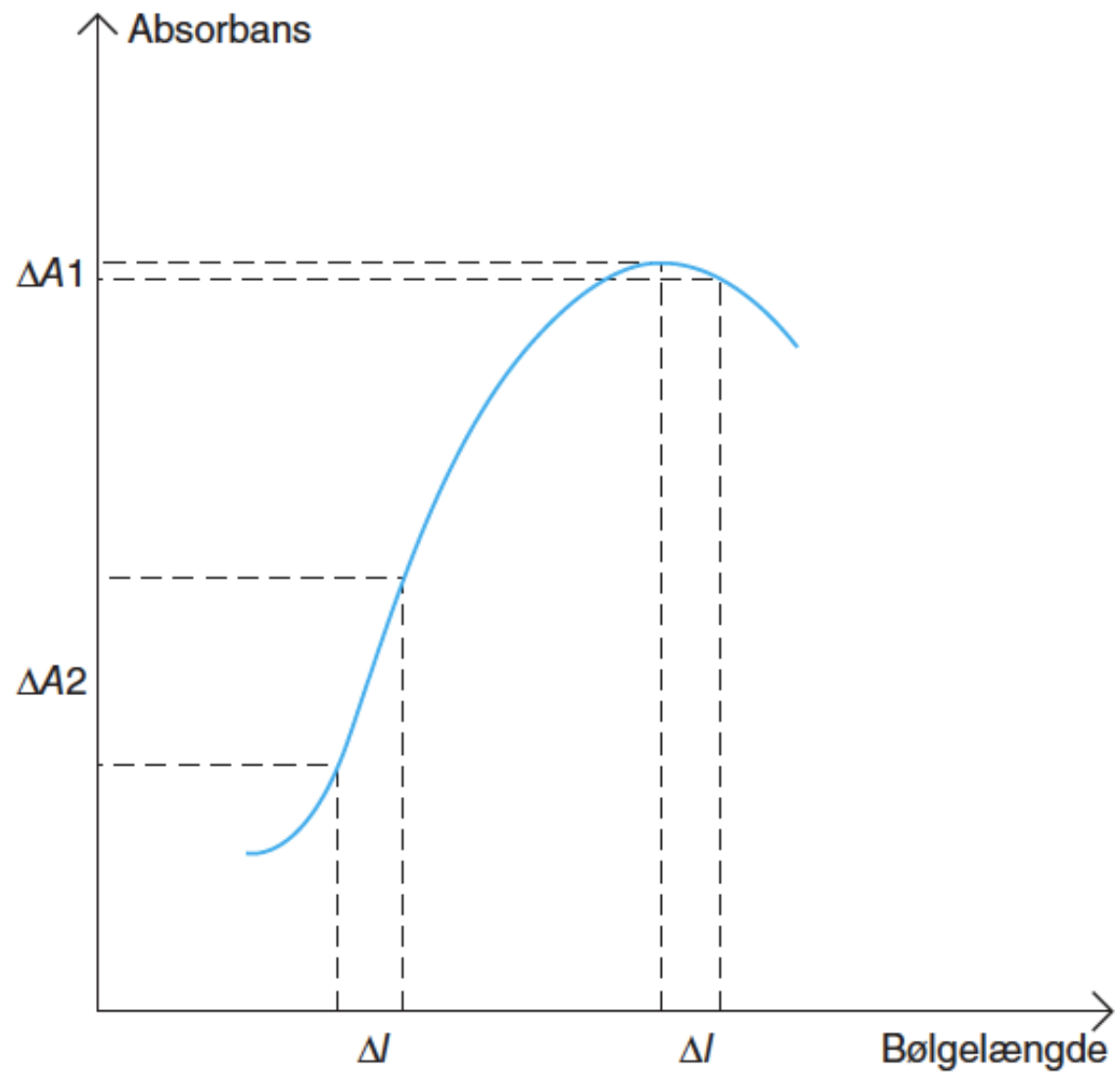
Figur 5.18. Nanodråbespektrofotometer.



Figur 5.19. Standardkurve.



Figur 5.20. Standardkurve, hvor linearitetsgrænsen er overskredet.



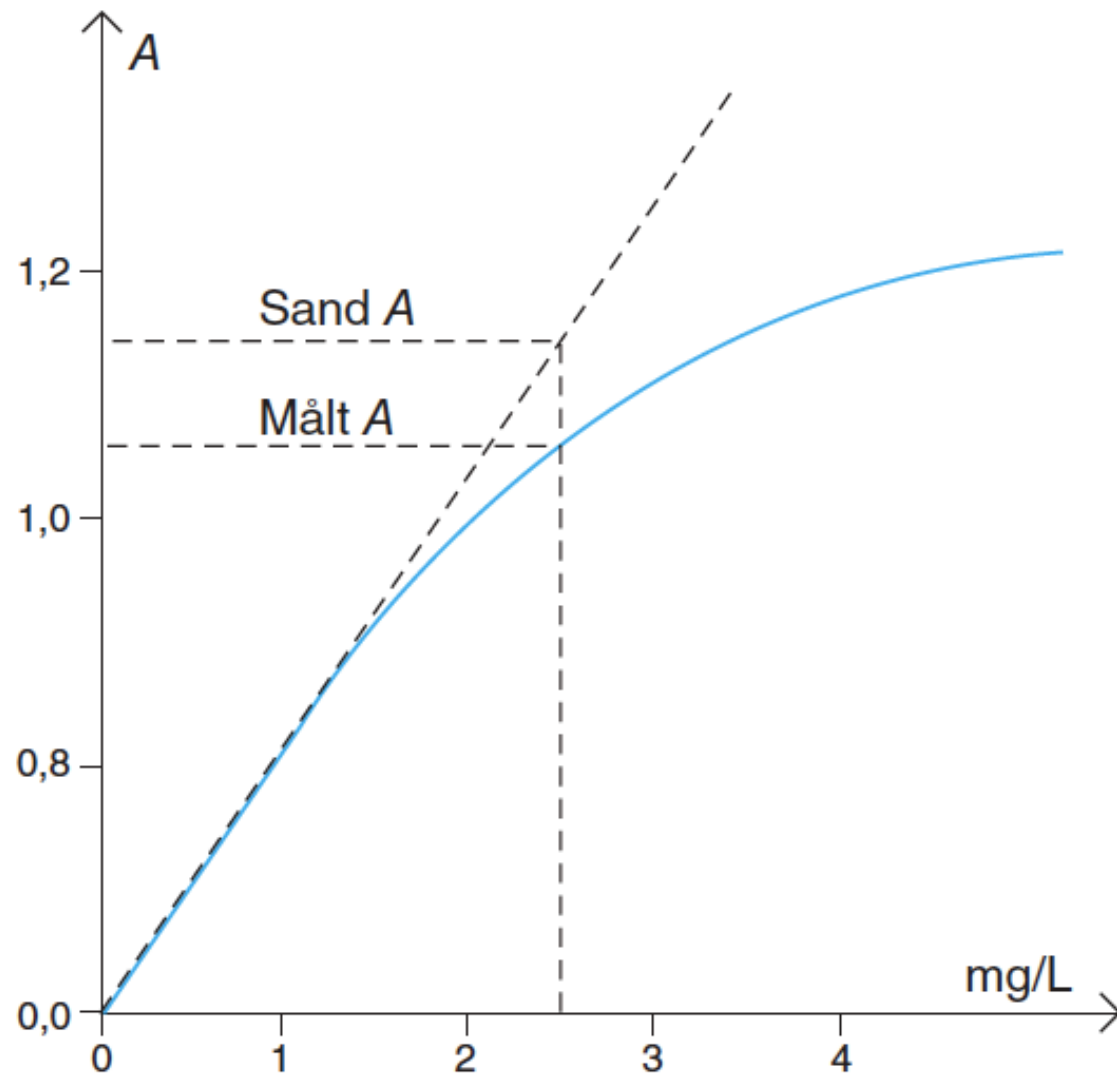
Figur 5.21. Bølgelængdenøjagtighed.

$\lambda_1 nm$	$\lambda_2 nm$	$\lambda_3 nm$	$\lambda_4 nm$	$\lambda_5 nm$
279,35	360,90	453,60	536,40	637,40
$\pm 0,25$	$\pm 0,25$	$\pm 0,25$	$\pm 0,25$	$\pm 0,5$

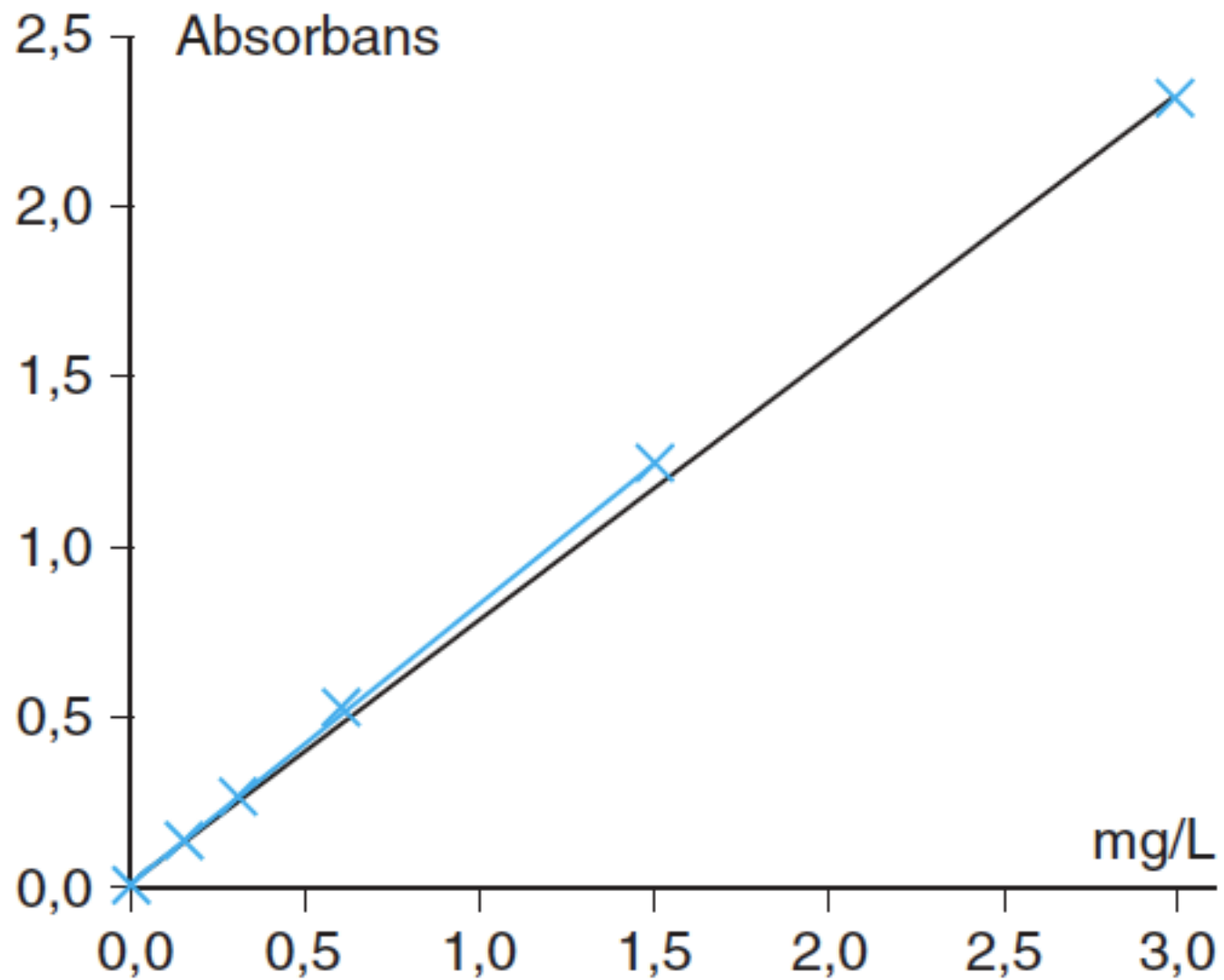
Tabel 5.2. Absorbansmaksimum for holmiumfilter.

λ	<i>stof</i>	<i>konc. i vand</i>
200 nm	NaBr	10 g/L
220 nm	NaI	10 g/L
340 nm	NaNO ₂	50 g/L
650 nm	Methylenblåt	0,01 % w/v

Tabel 5.3.



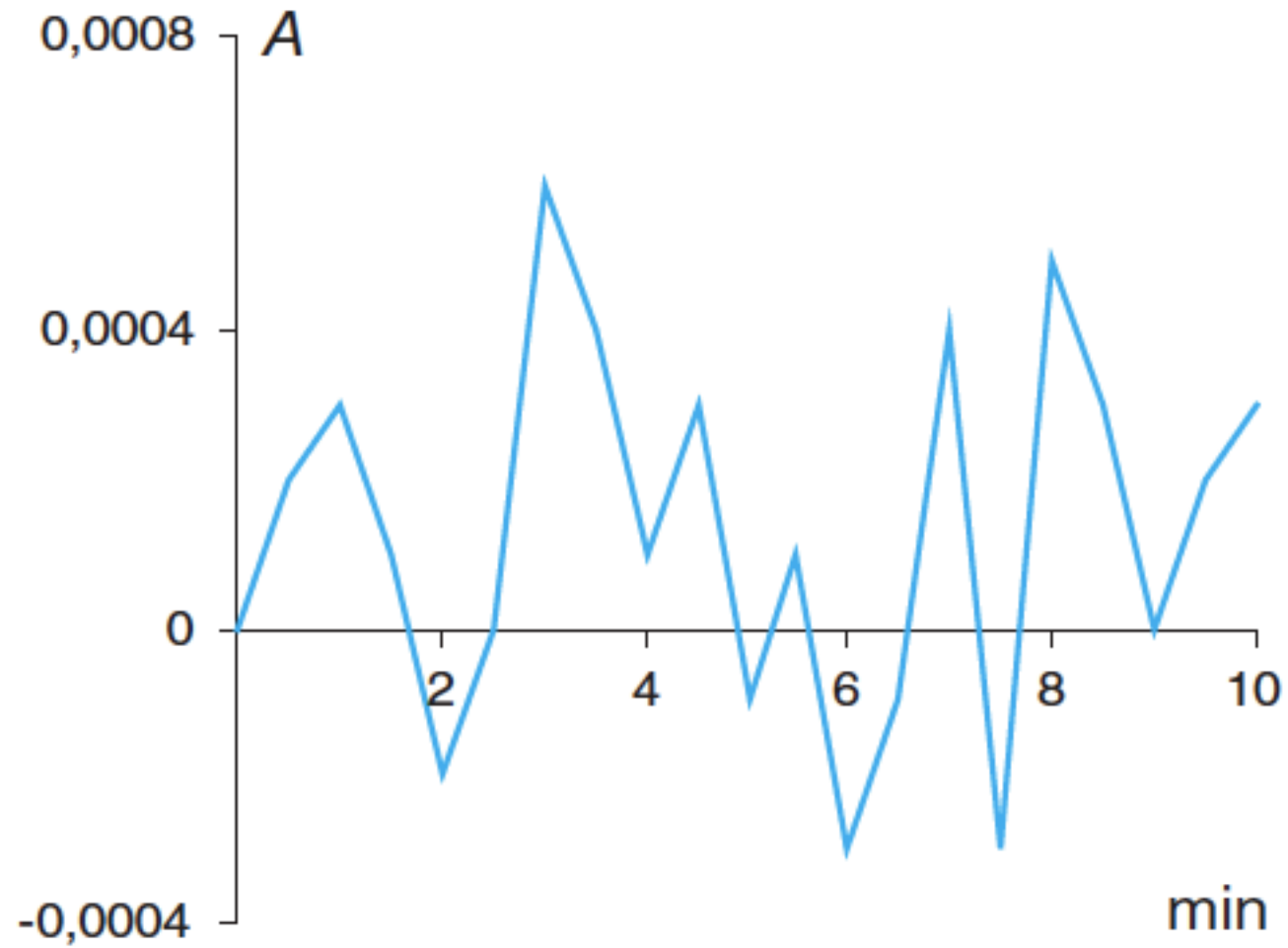
Figur 5.22. Stray light, der ikke absorberes i prøven.



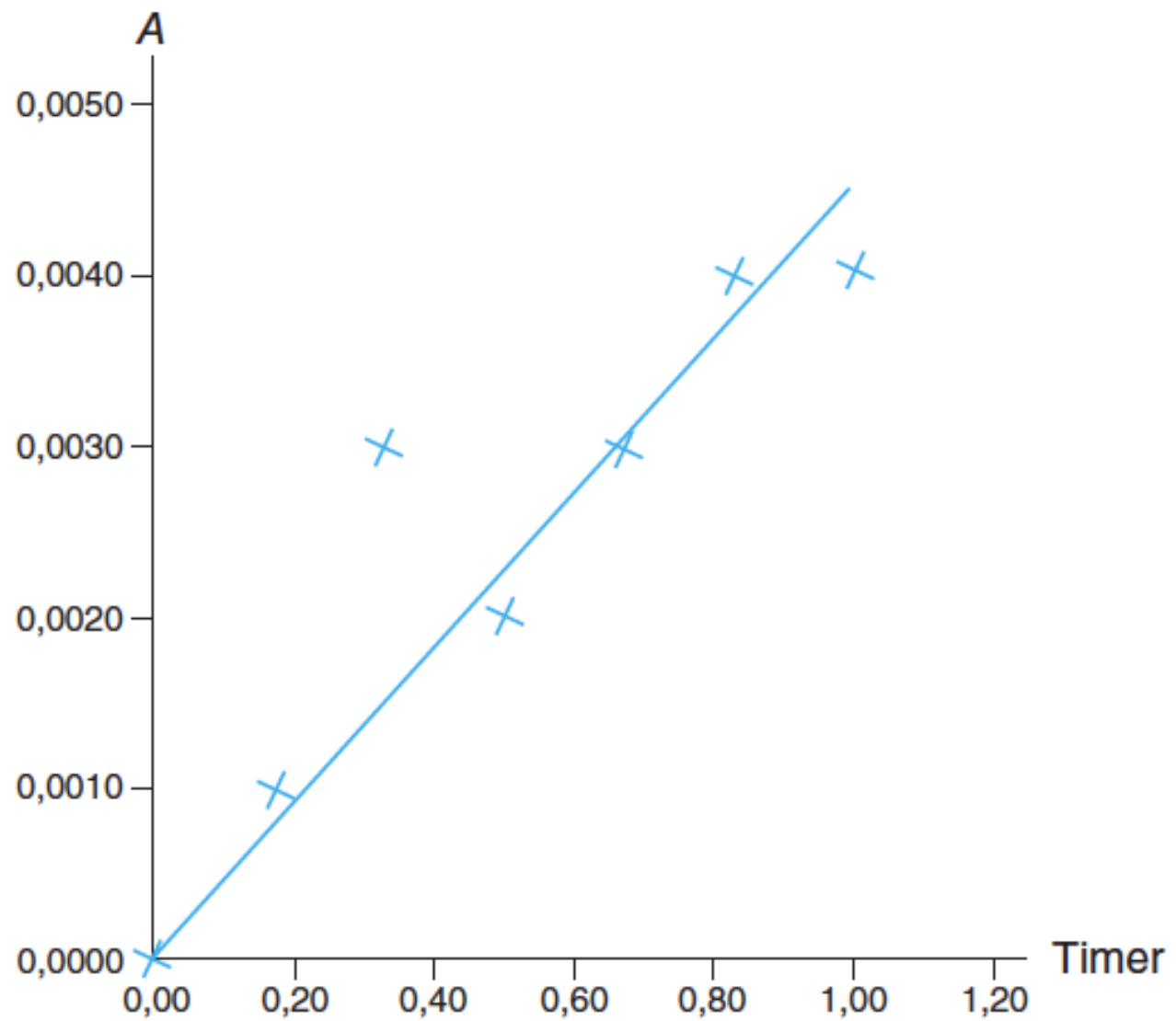
Figur 5.23. Linearitetsbestemmelse.

<i>Instrument</i>	<i>Støjkilder</i>	<i>Absorbansområde med minimal støj</i>
Billig type	Forstærker Detektor (solid state)	$0,4 < A < 0,8$
Kvalitetstype	Detektor (shut noise)	$0,4 < A < 2,0$

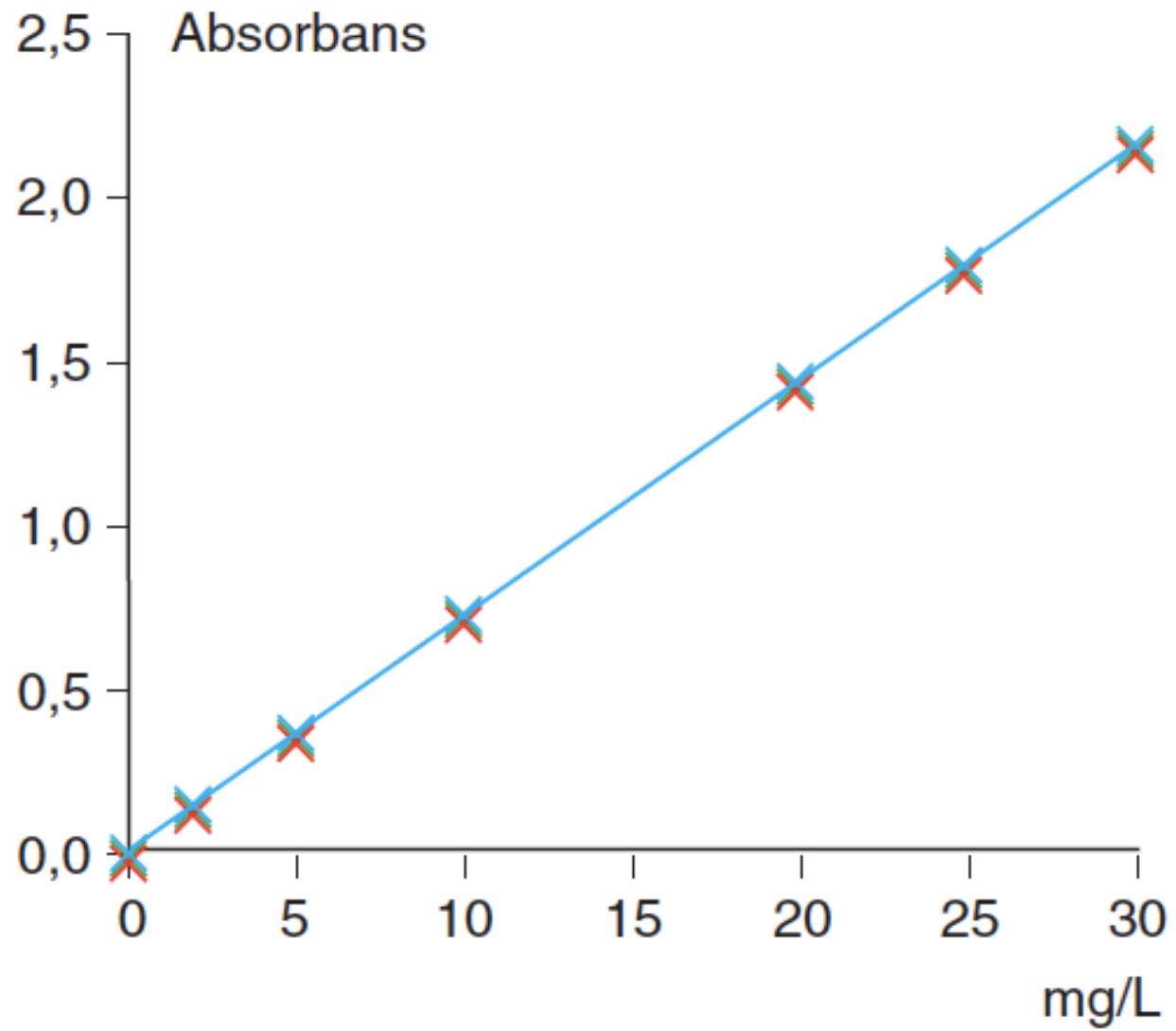
Tabel 5.4.



Figur 5.24. Bestemmelse af fotometerstøj.

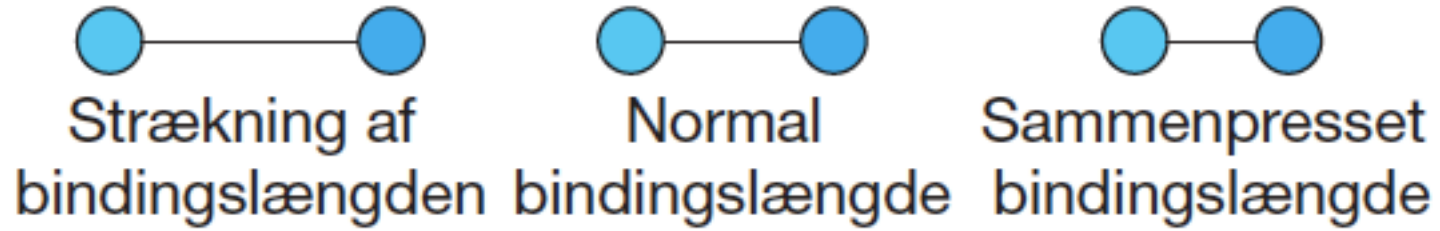


Figur 5.25. Bestemmelse af fotometerdrift.

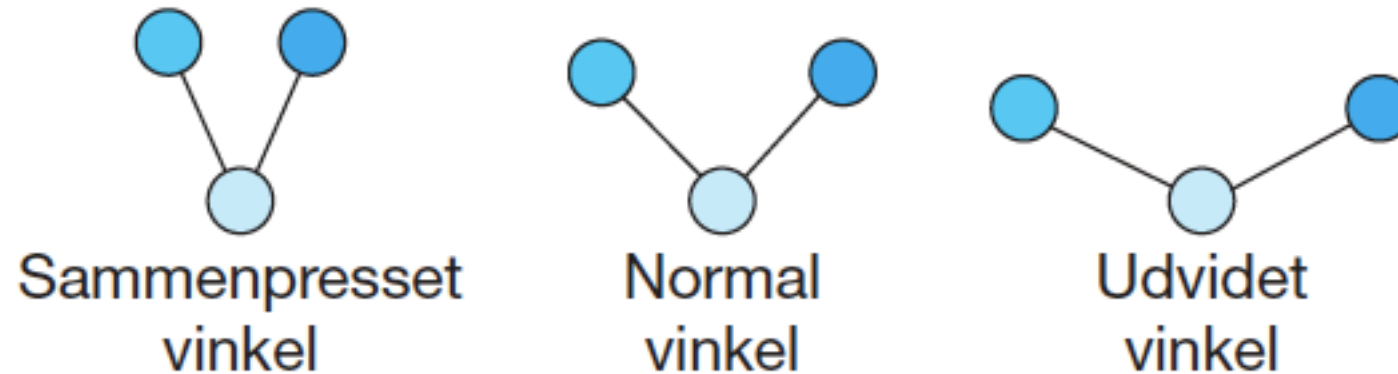


Figur 5.26. Validering af linearitet.

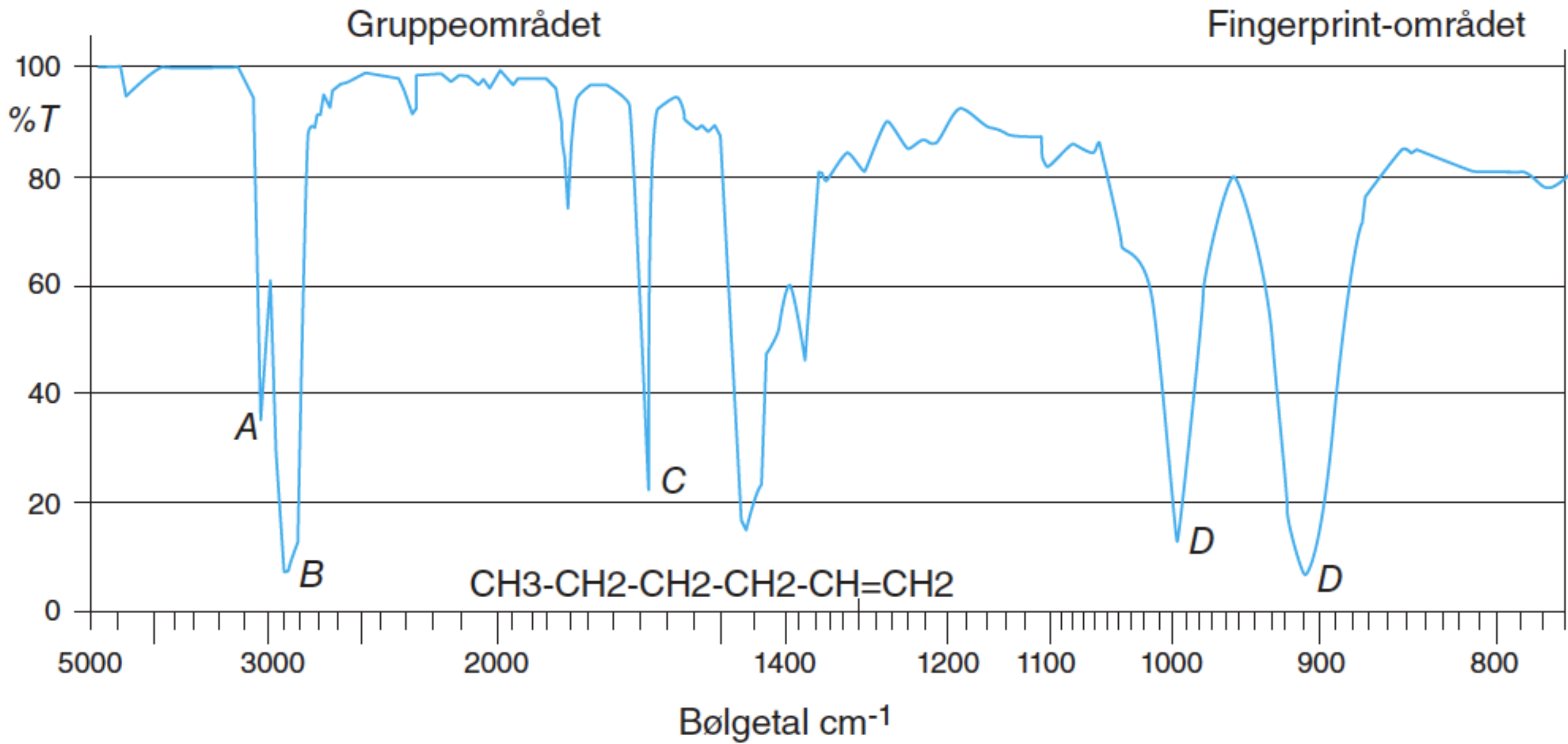
Strækning:



Bøjning:



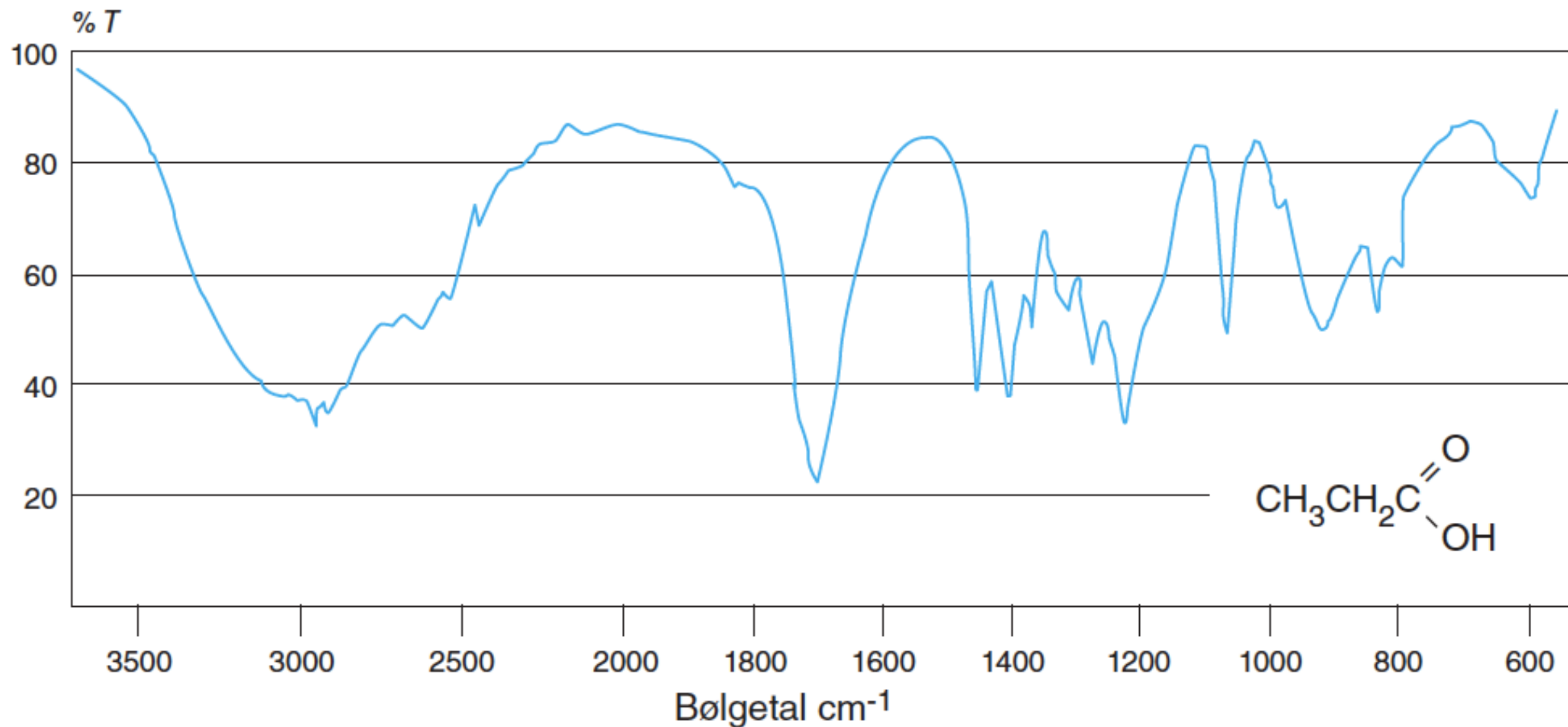
Figur 6.1. Vibrationsenergi og molekylbevægelse.



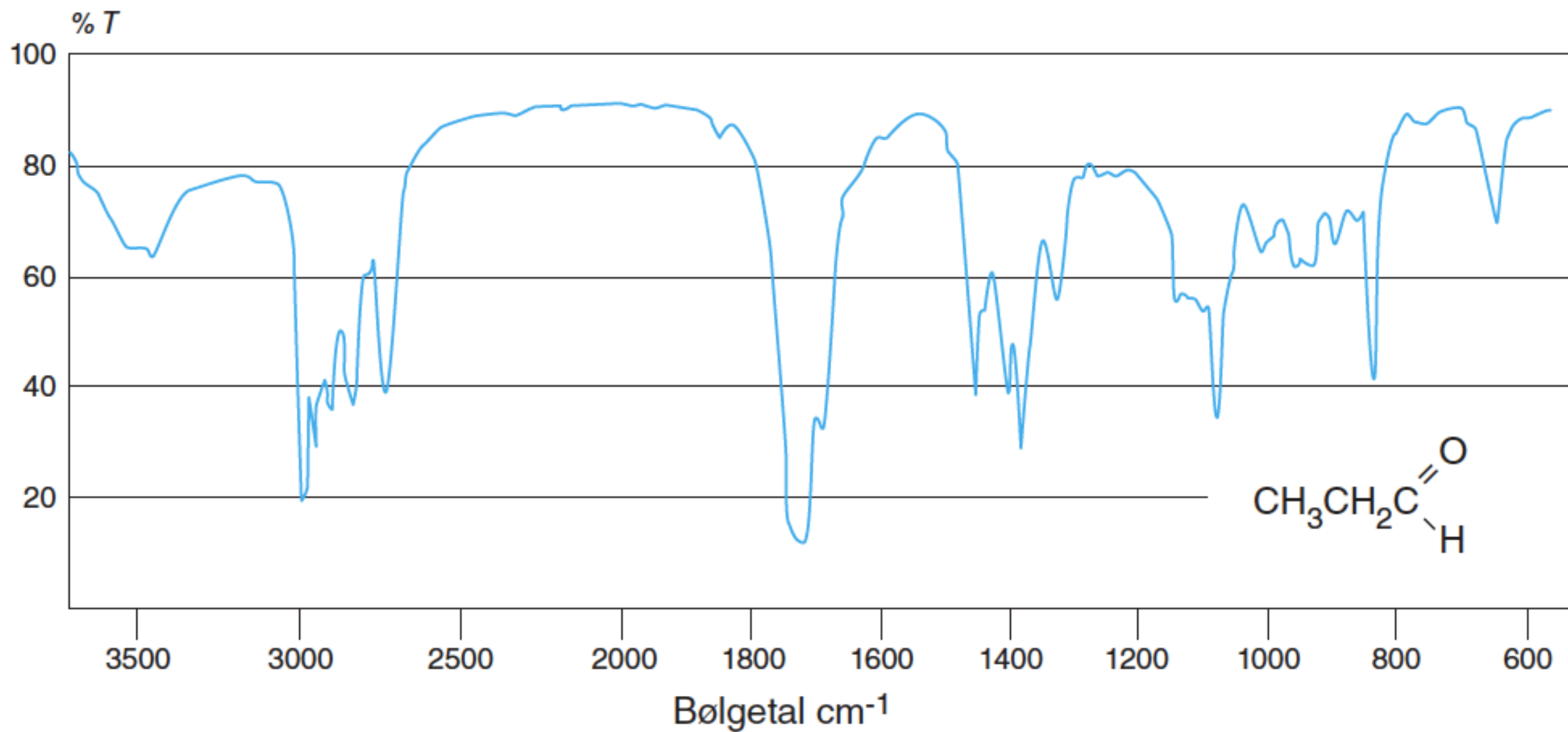
Figur 6.2. IR-spektrum af hexen.

<i>Binding</i>	<i>Forbindelse</i>	<i>Område (cm⁻¹)</i>
C=O	carbonylgruppe	1680-1760
C=O	carboxylsyre, ester	1700-1725
C=O	aldehyd	1720-1740
C=O	keton	1705-1725
C-O	carboxylsyre, ester	1050-1300
C-O	alkohol	1000-1300
C-O-C	ether	1000-1300
O-H	alkohol	3200-3600
O-H	carboxylsyre	2500-3000
C=C	alken	1610-1680
C=C	benzenring	1500-1600
C-H	aldehyd	2750 og 2850
C-H	alken	3010-3100
C-H	benzenring	3010-3100
C-H	alkan	2850-3000
		1340-1470
C-N	amin/amid	1180-1360
N-H	amin/amid	3300-3500
		1580-1650

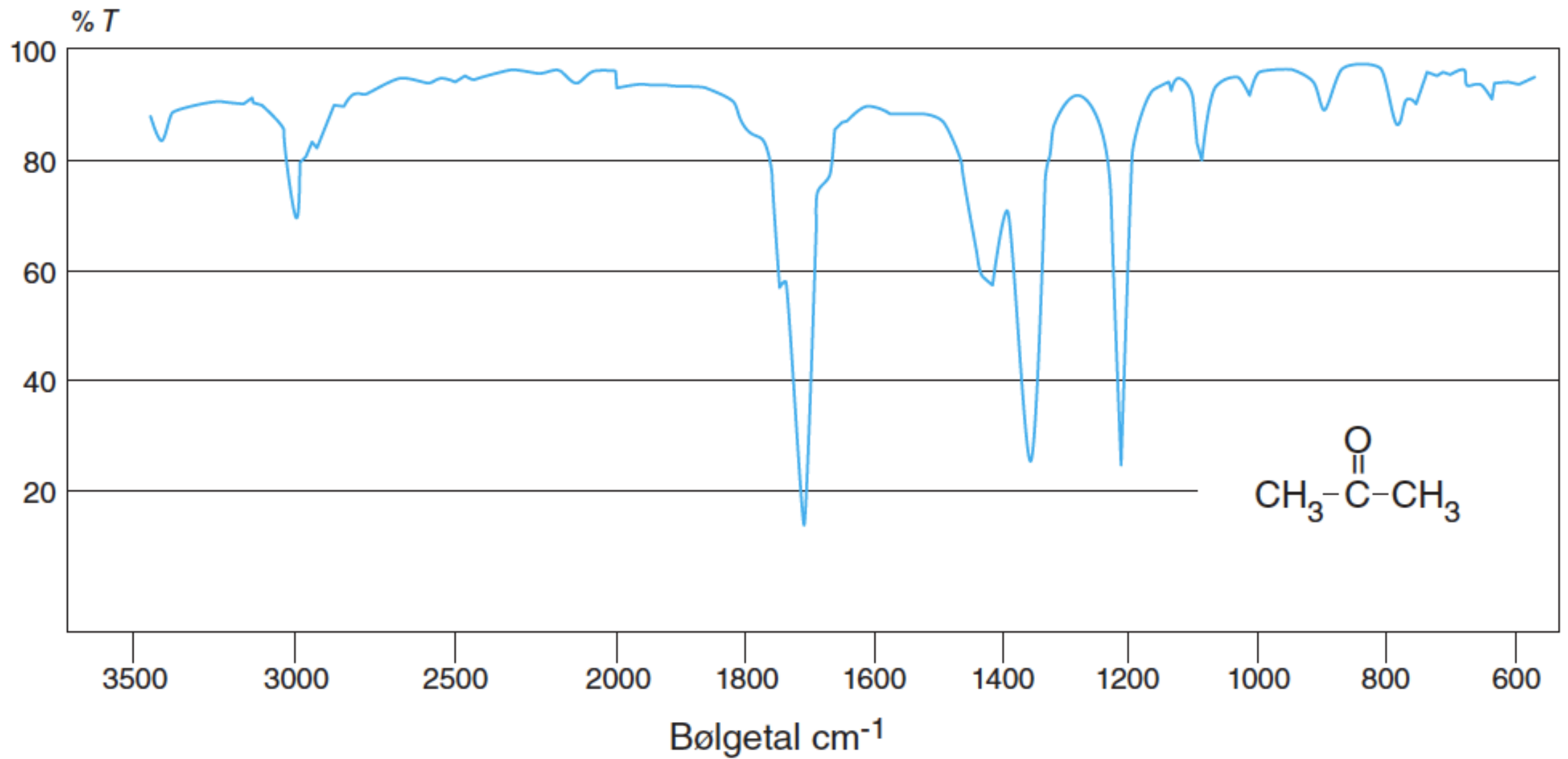
Tabel 6.1.



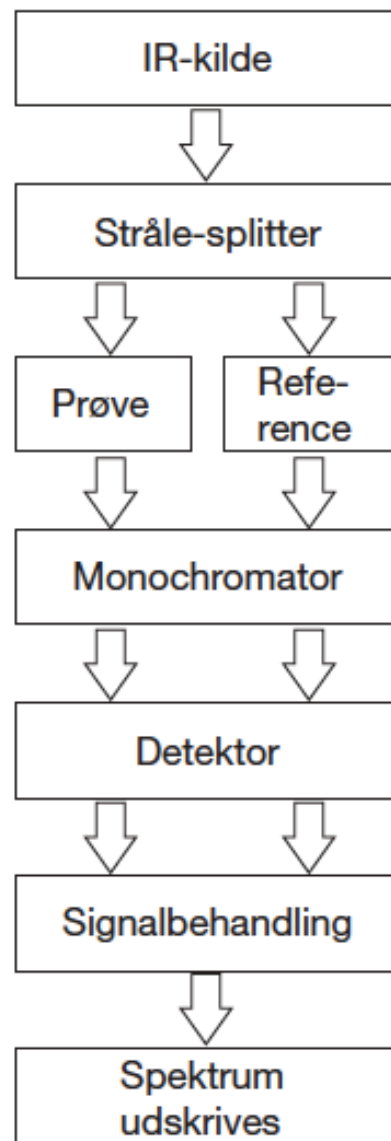
Figur 6.3. IR-spektrum for propansyre.



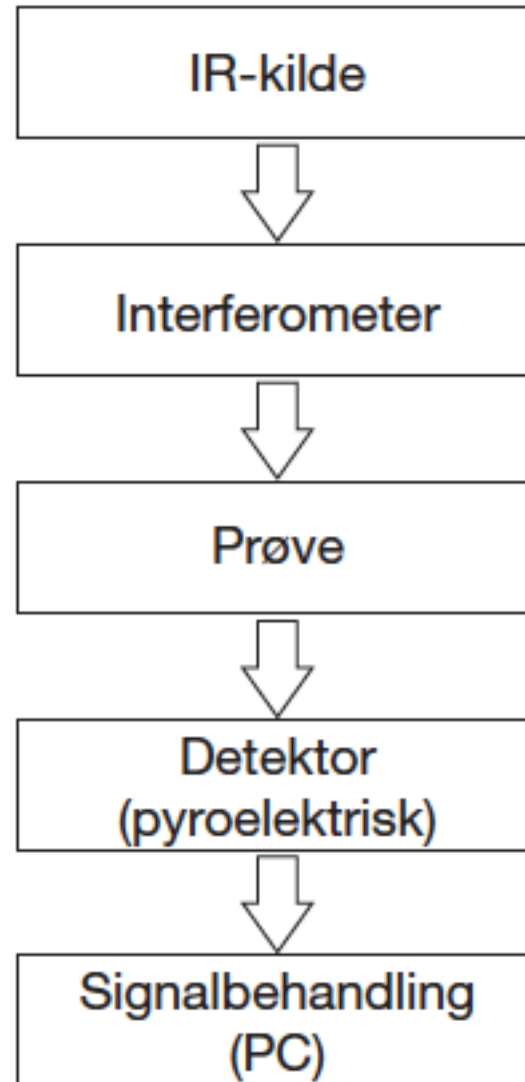
Figur 6.4. IR-spektrum for propanal.



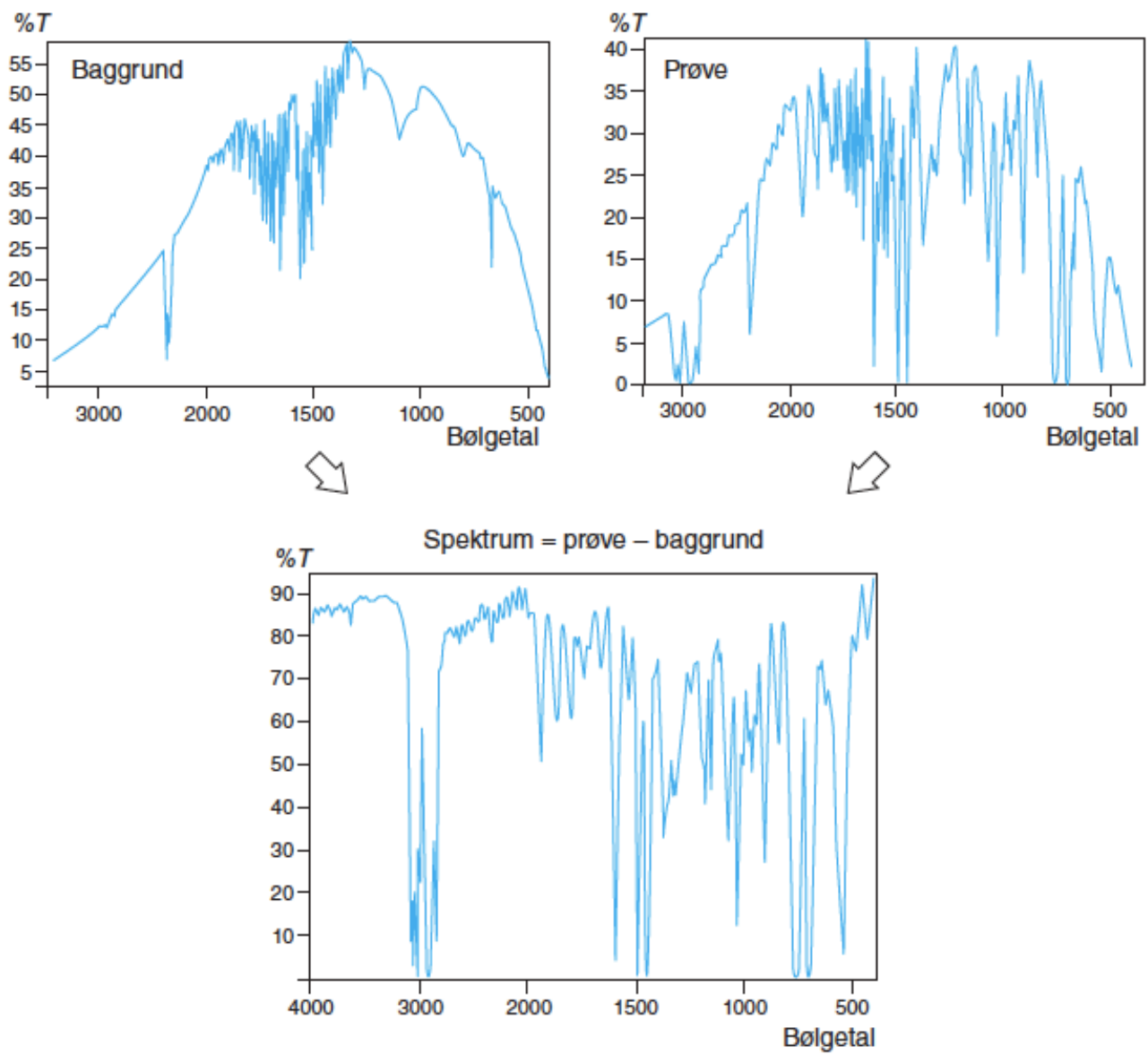
Figur 6.5. IR-spektrum for propanon.



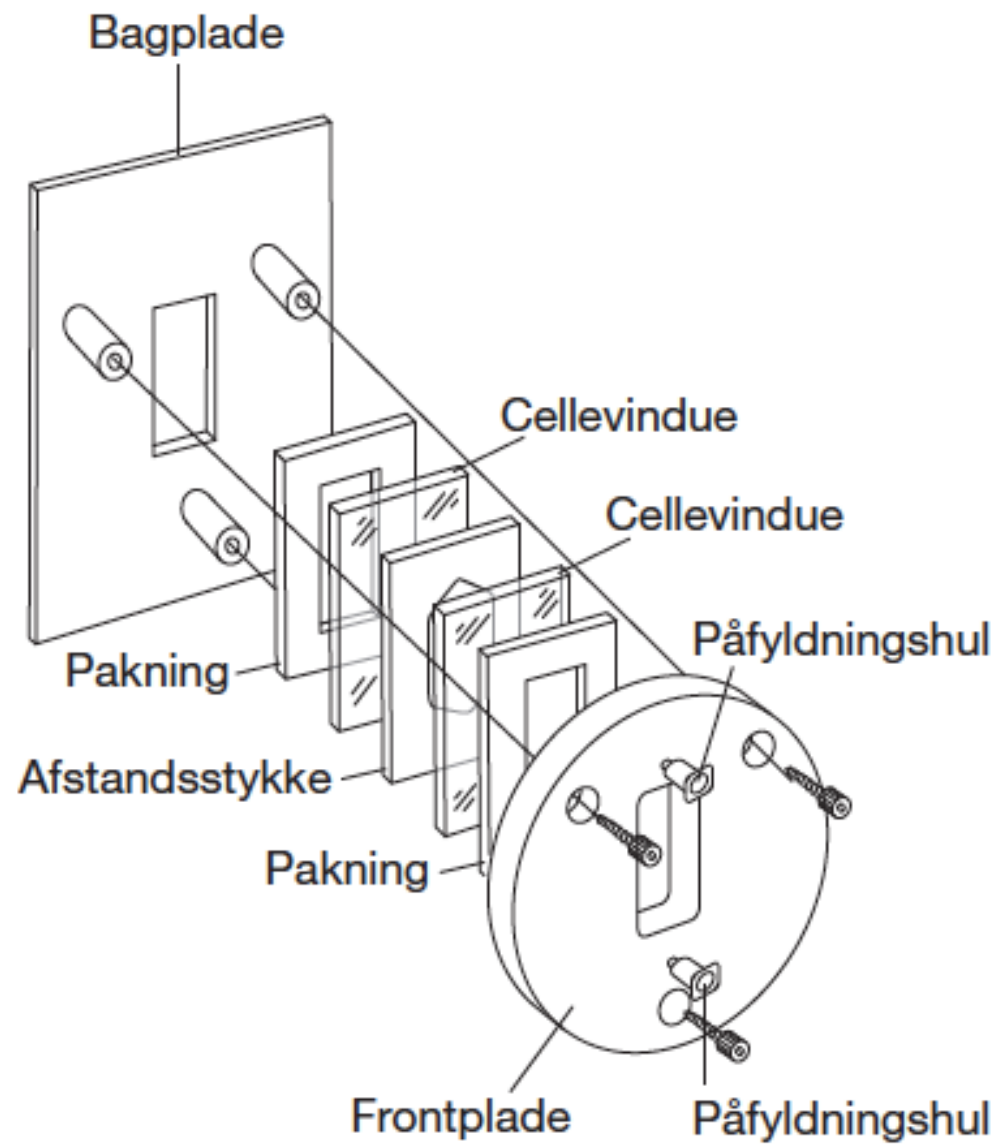
Figur 6.6. Ældre IR-spektrofotometers opbygning.



Figur 6.7. Opbygningen af et FT-IR instrument.



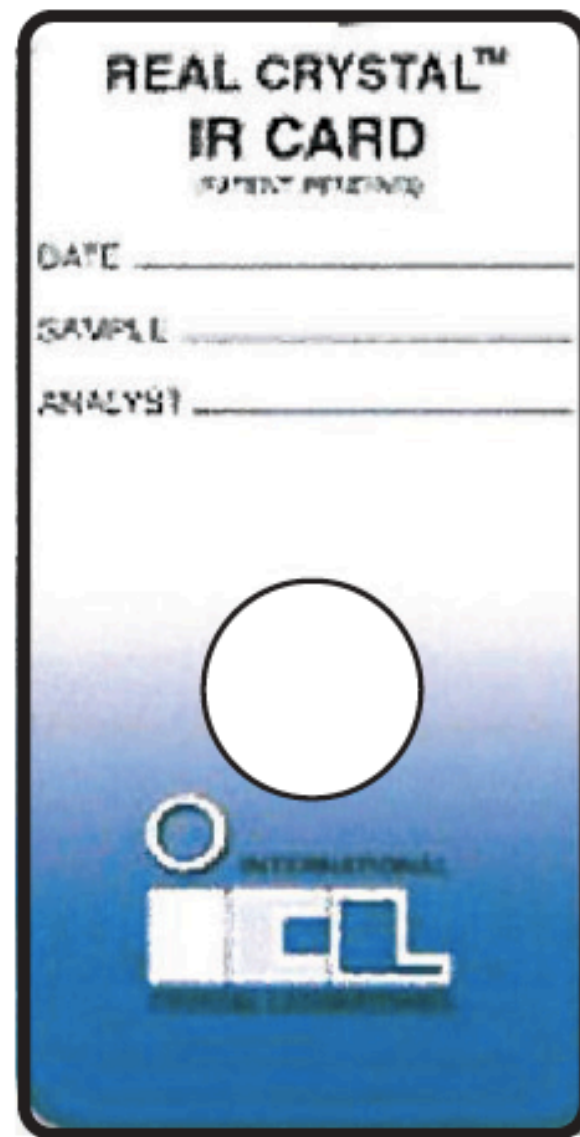
Figur 6.8. Spektrum af baggrund og prøve samt det endelige IR-spektrum af prøven, hvor baggrundsspektret er fratrukket.



Figur 6.9. Samling af væskekuvette.

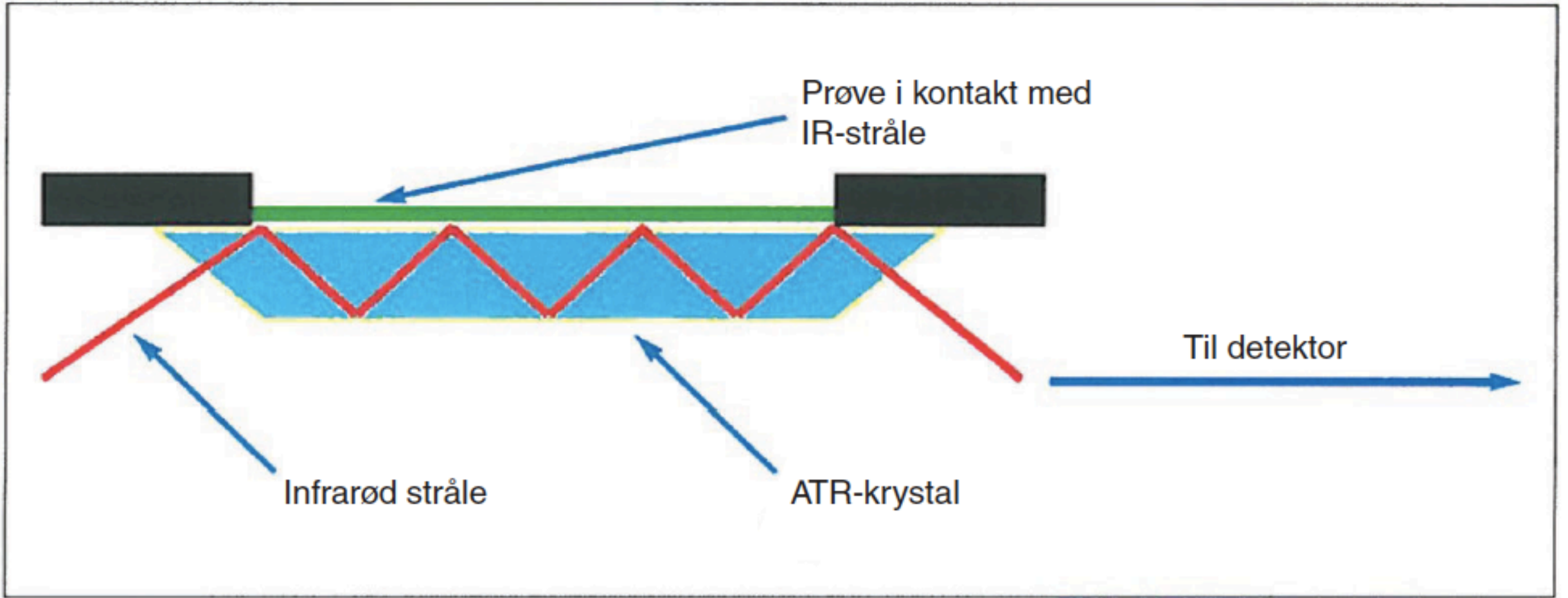
<i>Materiale</i>	<i>Område (cm⁻¹)</i>	<i>Bemærk</i>
CaF ₂	4000-1100	Kan bruges med vand
NaCl	4000-625	Hygroskopisk
KBr	4000-375	Hygroskopisk
KR5 (TlBr-TlI)	4000-90	Kan bruges med vand, meget blødt

Tabel 6.2.

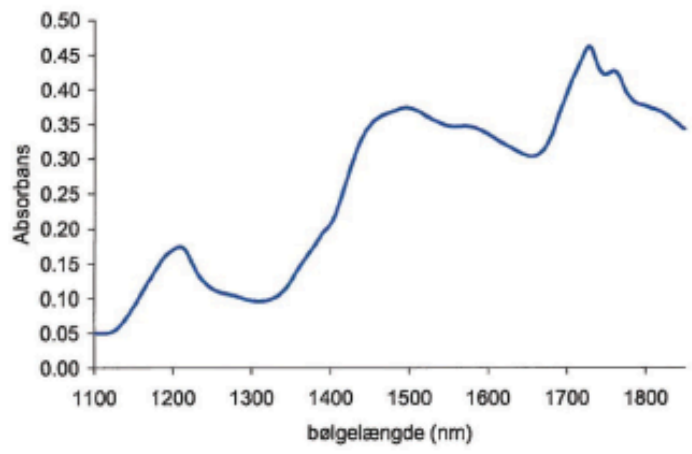


Figur 6.10. Papkort med indbygget rude til IR-måling.

ATR



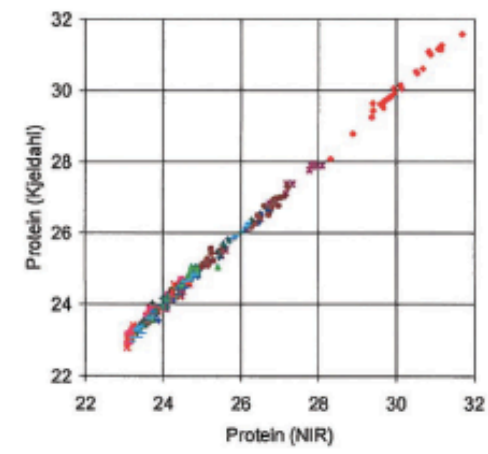
Figur 6.11. Princippet i ATR (efter Perkin Elmer).



8300 NIR spektre af mælkepulver

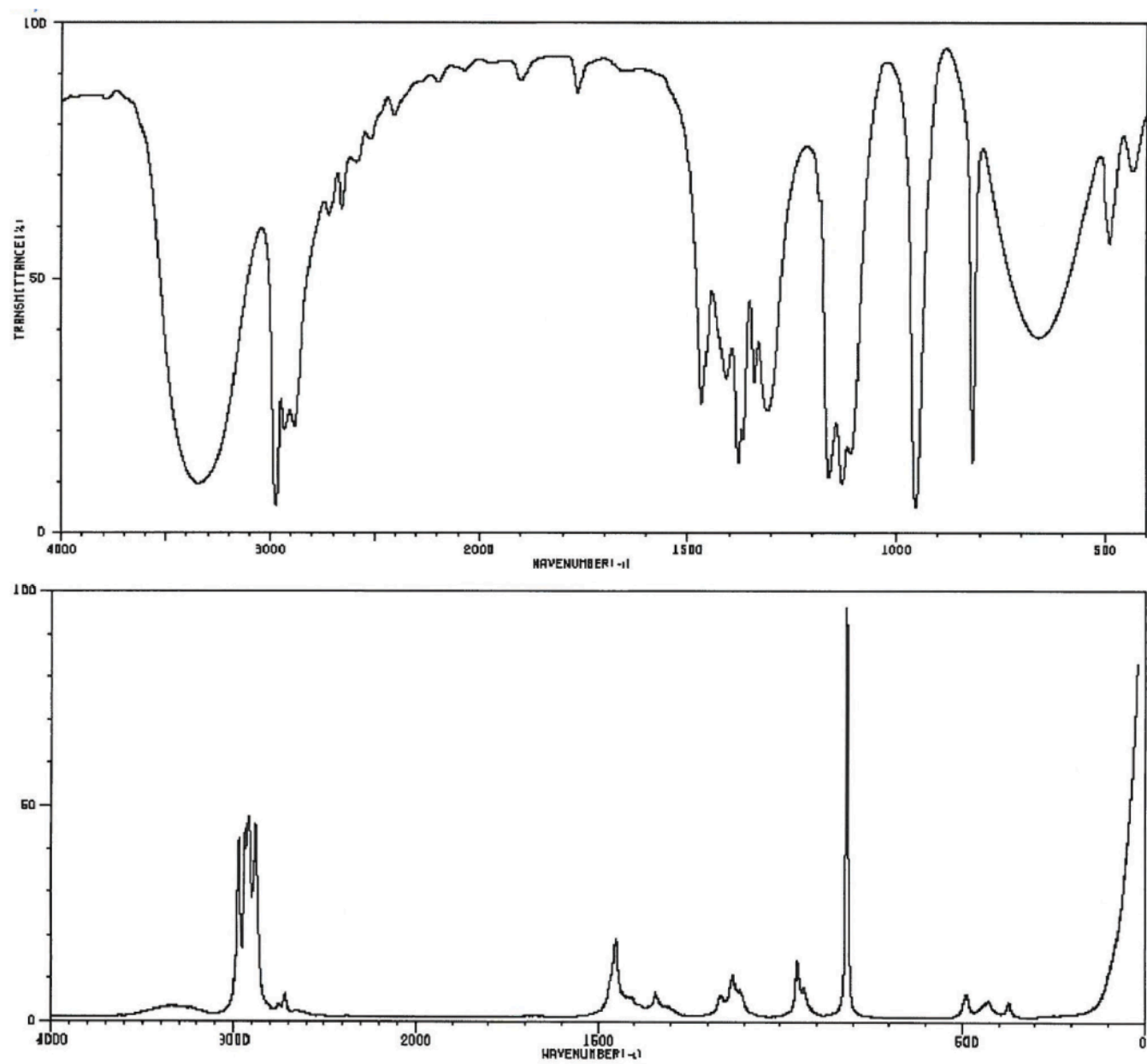


8300 Kjeldahl analyser

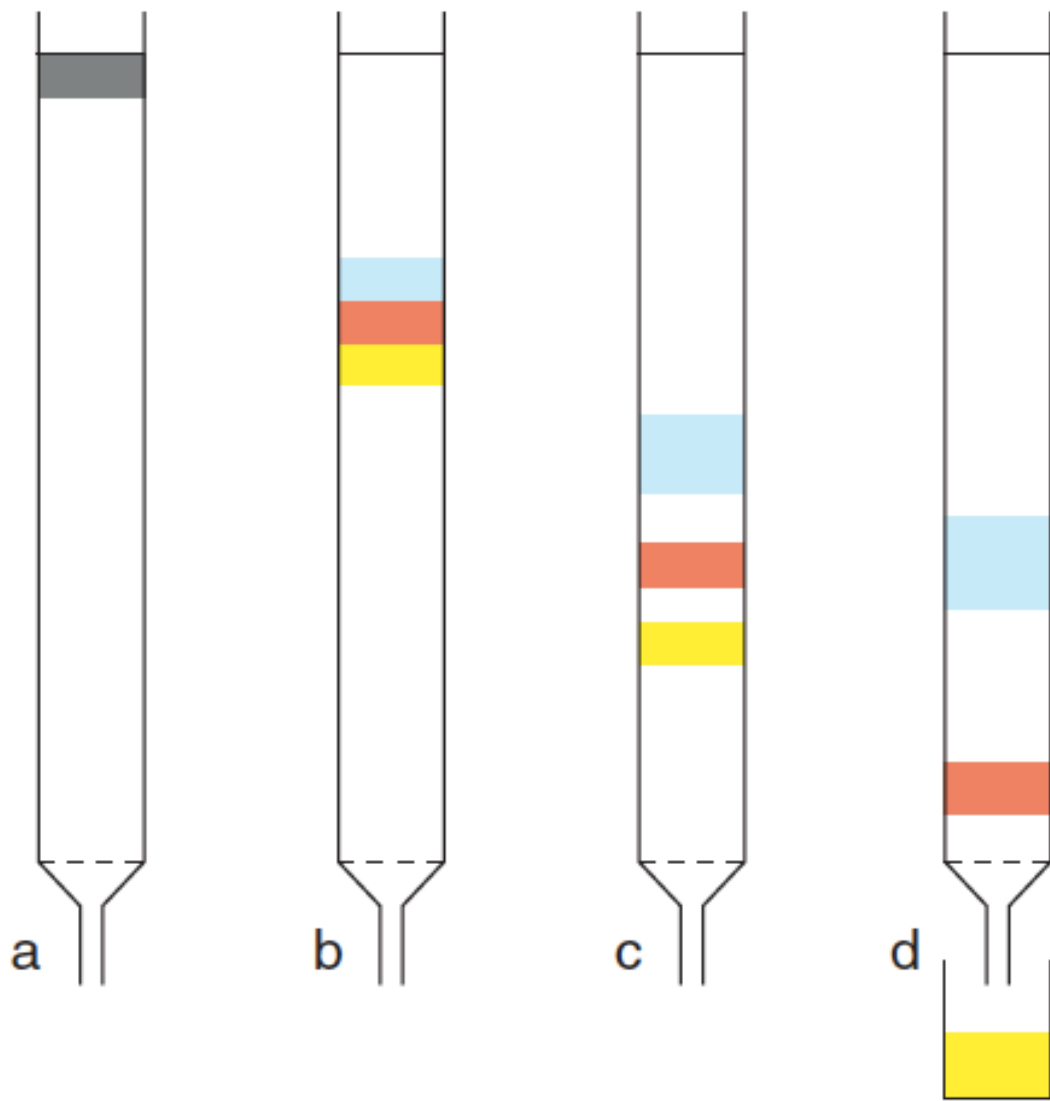


Nøjagtighed: 0.11

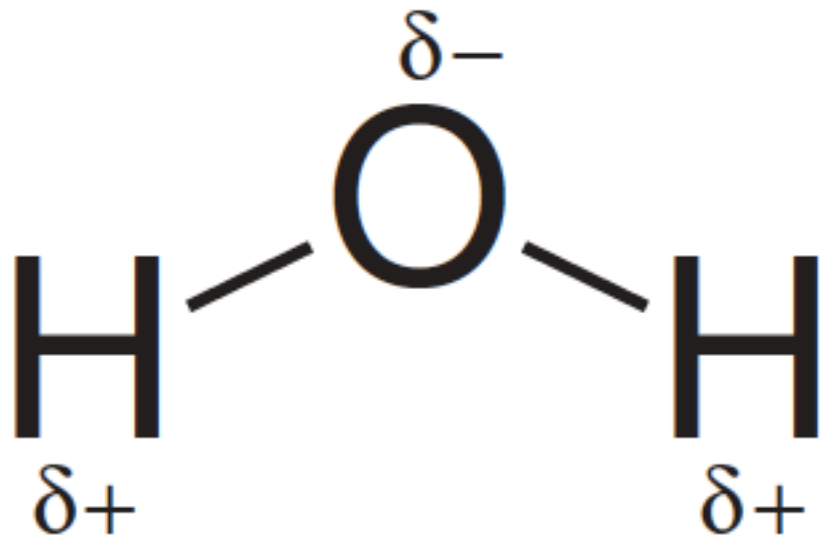
Figur 6.12. Sammenhængen mellem NIR-målinger og en klassisk vådkemisk bestemmelse af protein i mælkepulver.



Figur 6.13. IR-spektrum (øverst) og Raman-spektrum (nederst) af propan-2-ol.



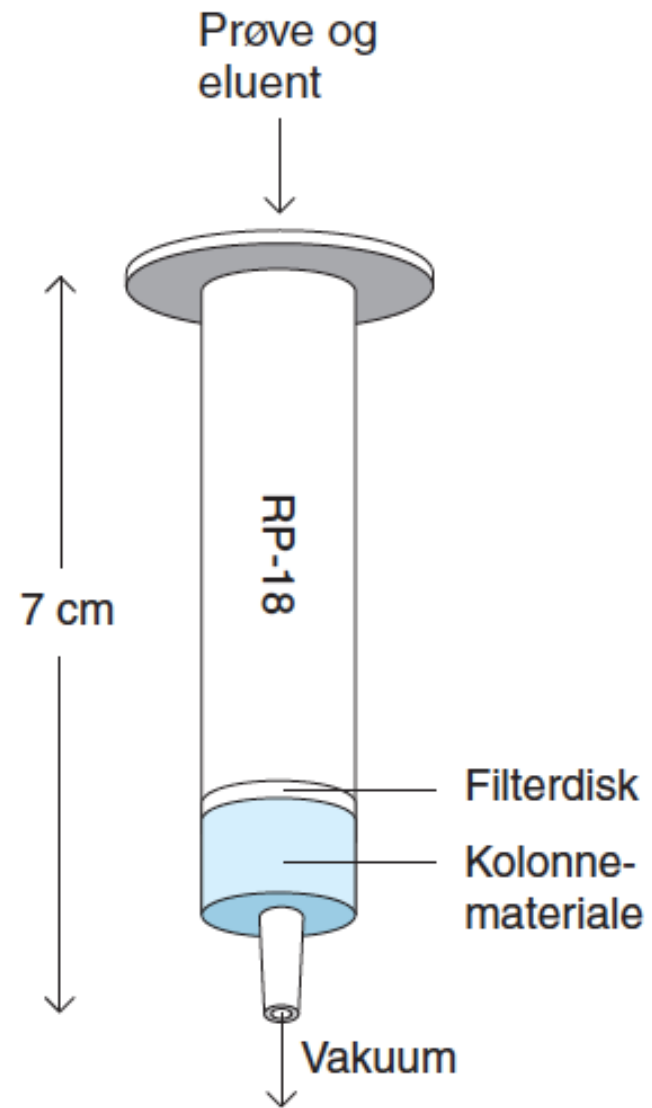
Figur 7.1. Søjlechromatografi.



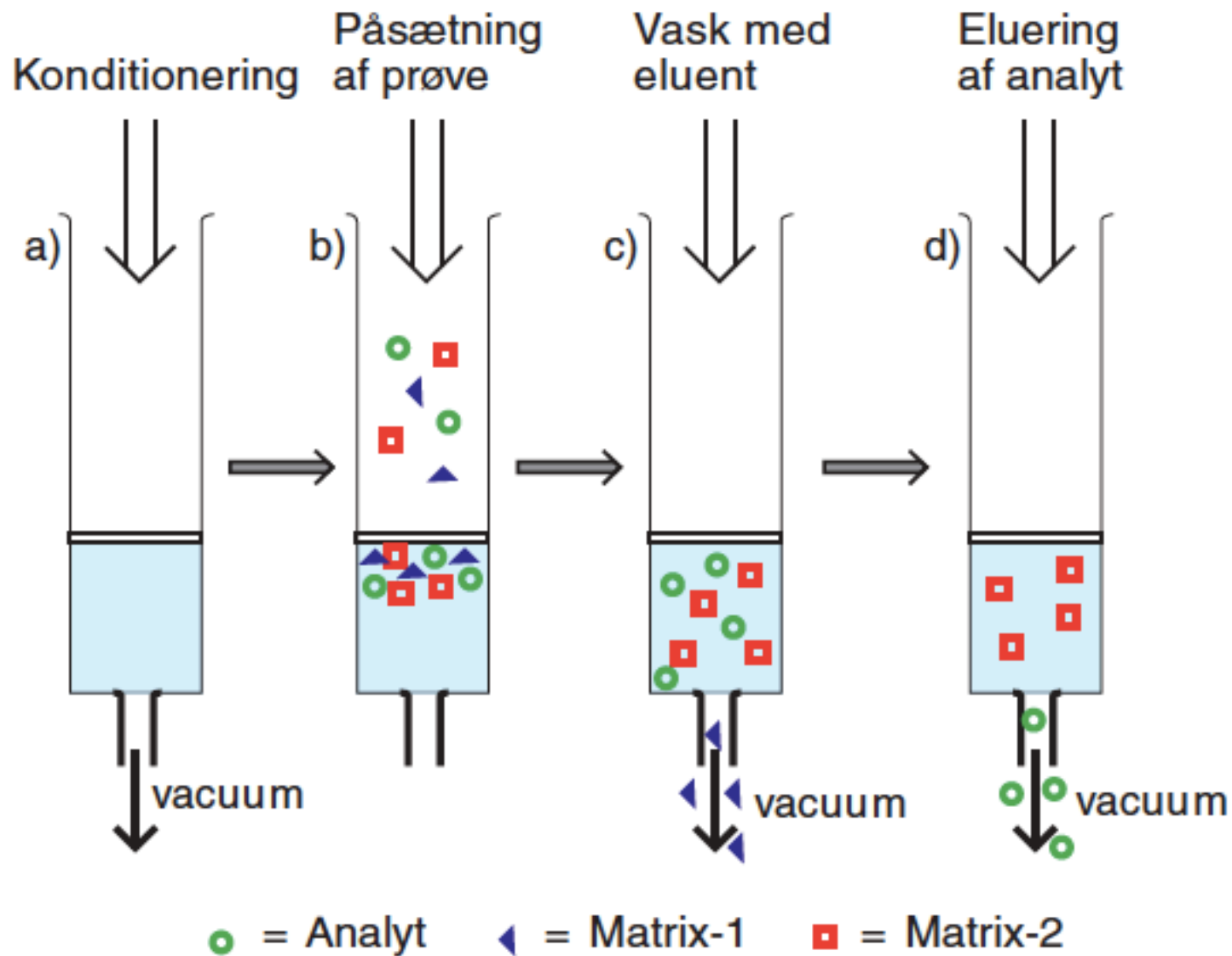
Figur 7.2. Vandmolekylets polarisering.

<i>Opløsningsmiddel</i>	<i>Elueringsstyrke⁸⁰ ε°</i>
n-hexan	0,01
Toluen	0,29
Methylenchlorid	0,42
Tetrahydrofuran	0,45
Methyl-ethyl keton	0,51
Acetone	0,56
Acetonitril	0,65
propan-2-ol	0,82
Ethanol	0,88
Methanol	0,95
Vand	stor

Tabel 7.1.



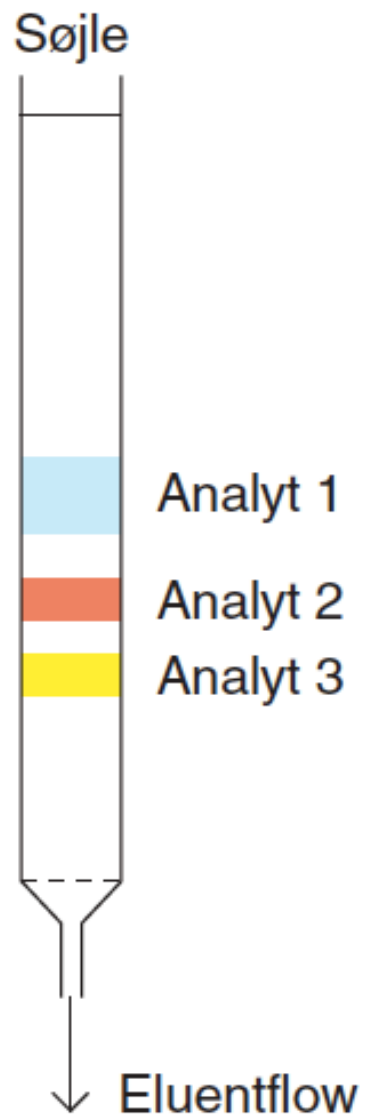
Figur 7.3. SPE kolonne.



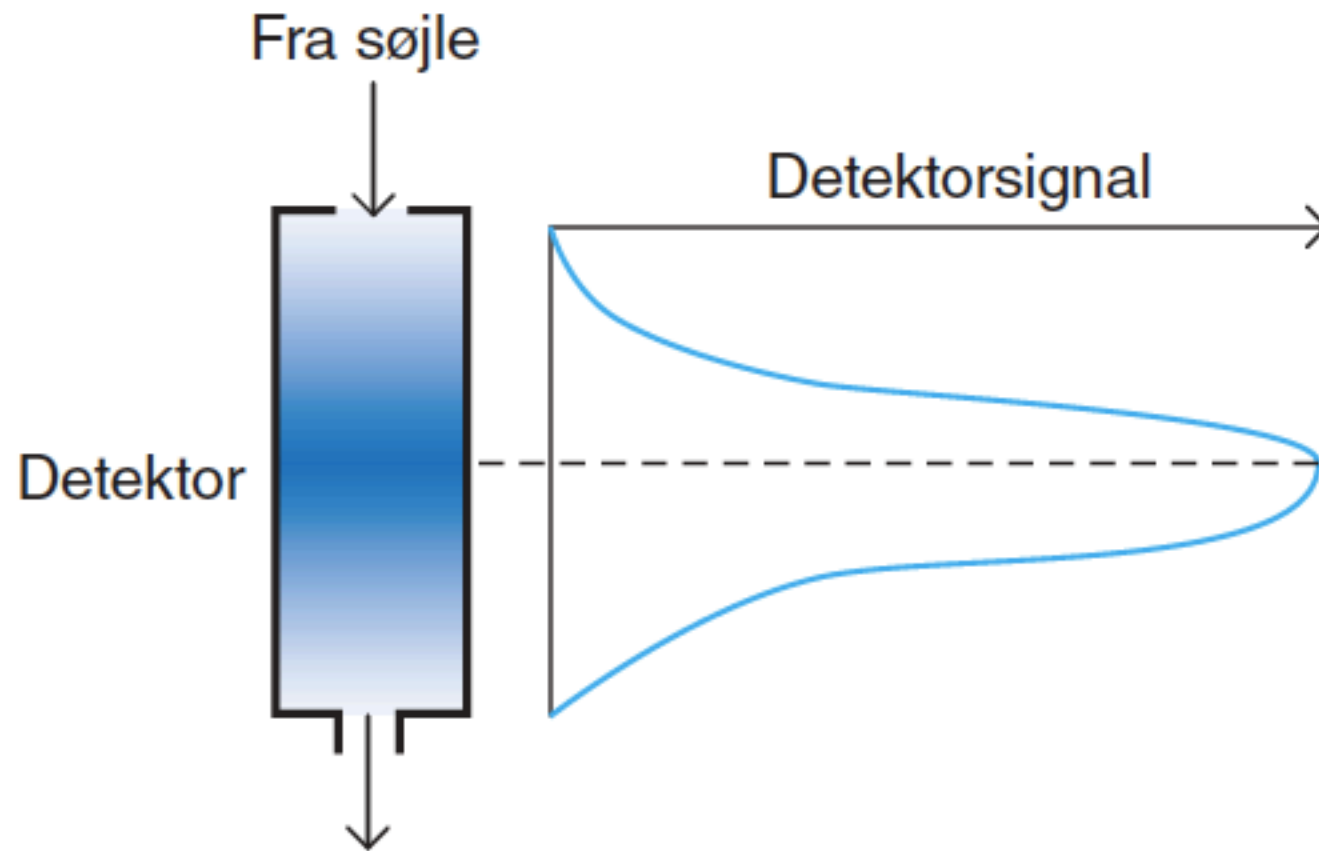
Figur 7.4. Fast fase ekstraktion, SPE.

<i>Kolonne</i>	<i>Prøvematrix</i>	<i>Eluent</i>	<i>Anvendelse</i>
C18	vandig	methanol acetonitril chloroform	medicinalvarer pesticider konserveringsmidler
Nitril	vandig	methanol acetonitril chloroform	medicinalvarer pesticider vitaminer
Amin	organisk	methanol propan-2-ol	vitaminer lipider kulhydrater phenoler

Tabel 7.2.

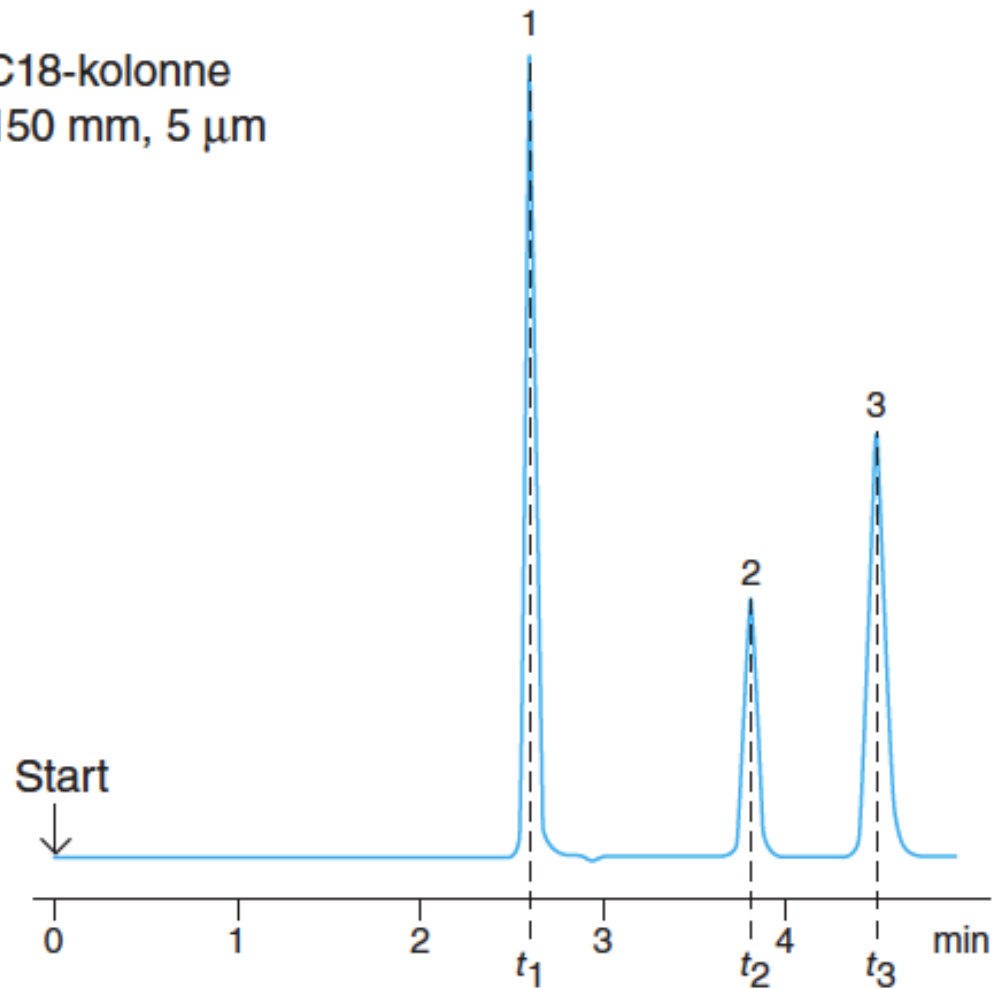


Figur 8.1. Adskillelse af prøvekomponenter på søjle.



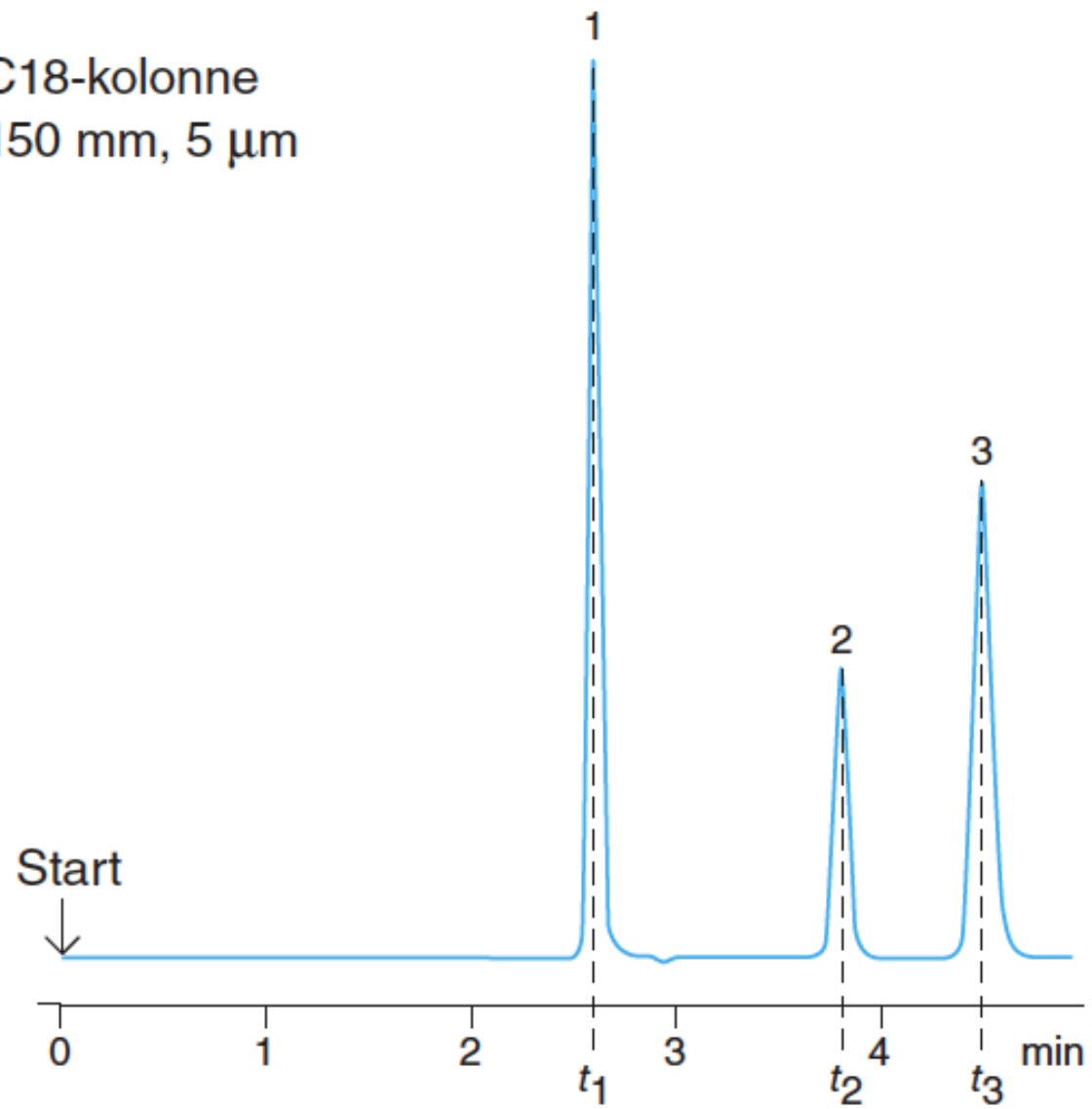
Figur 8.2. En af prøvens komponenter detekteres.

C18-kolonne
150 mm, 5 μm

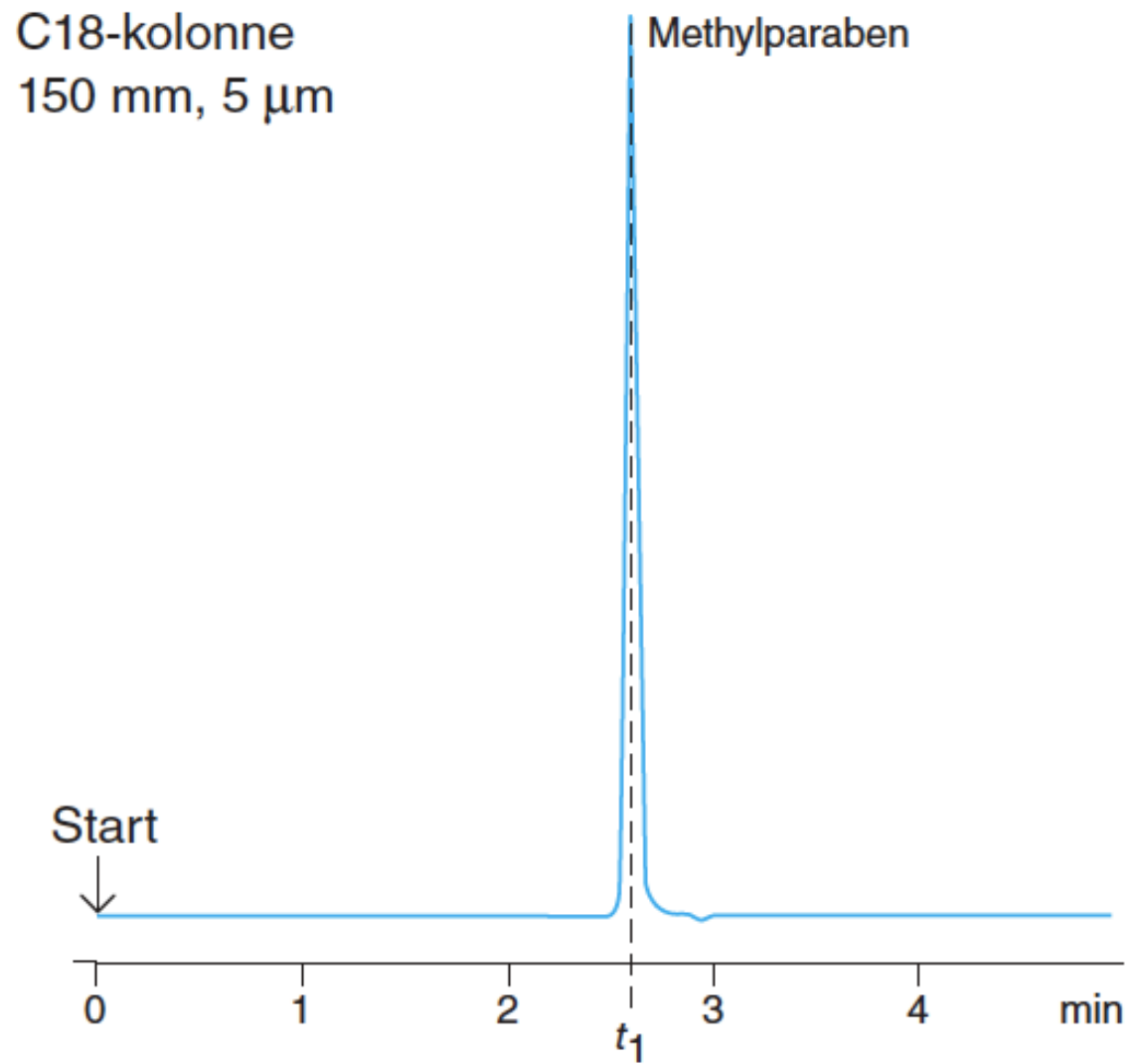


Figur 8.3. Chromatografering af prøve, der indeholder tre komponenter. Bemærk at toppenes bredde bliver lidt større jo længere tid, komponenterne har opholdt sig i systemet.

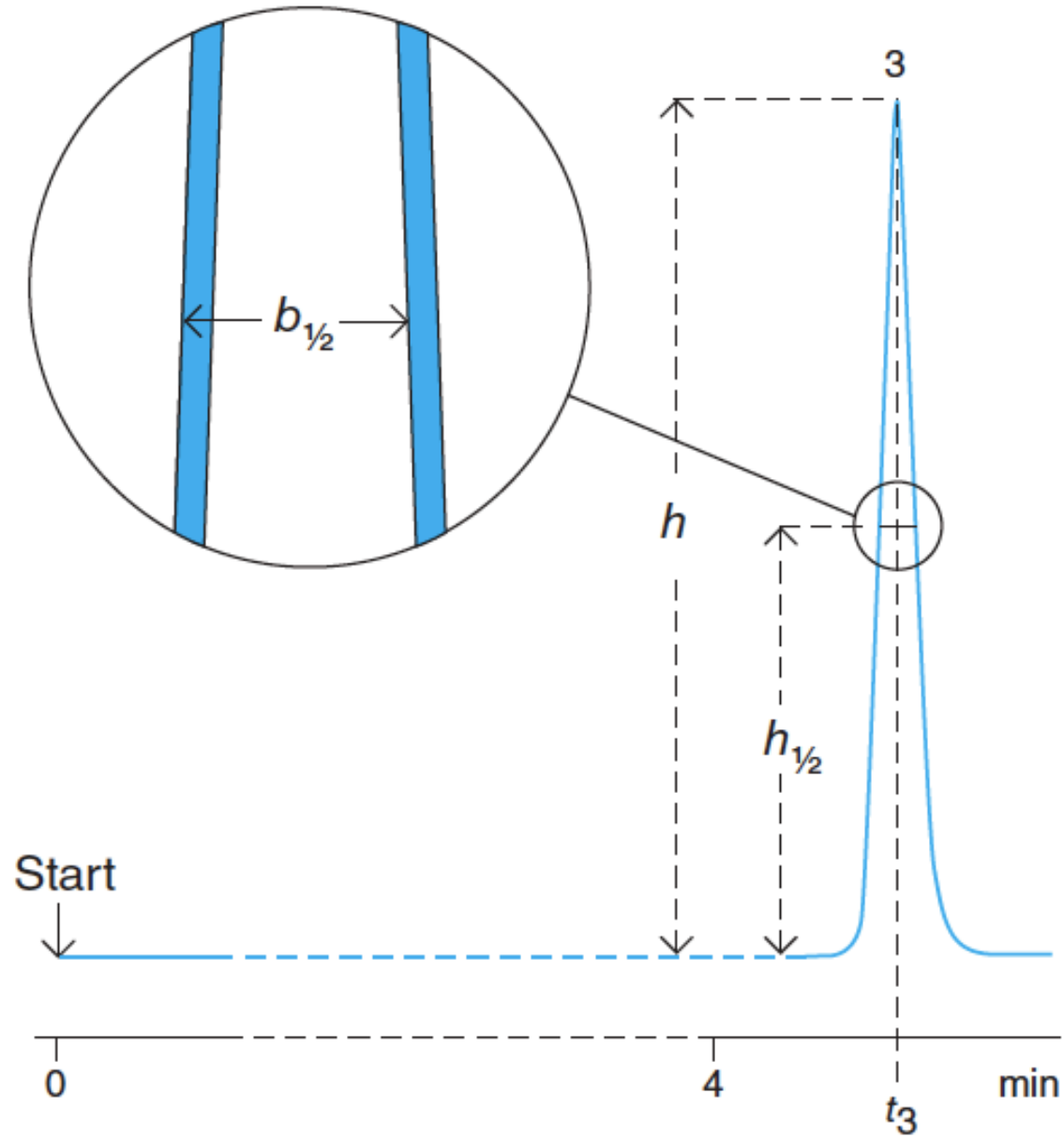
C18-kolonne
150 mm, 5 μm



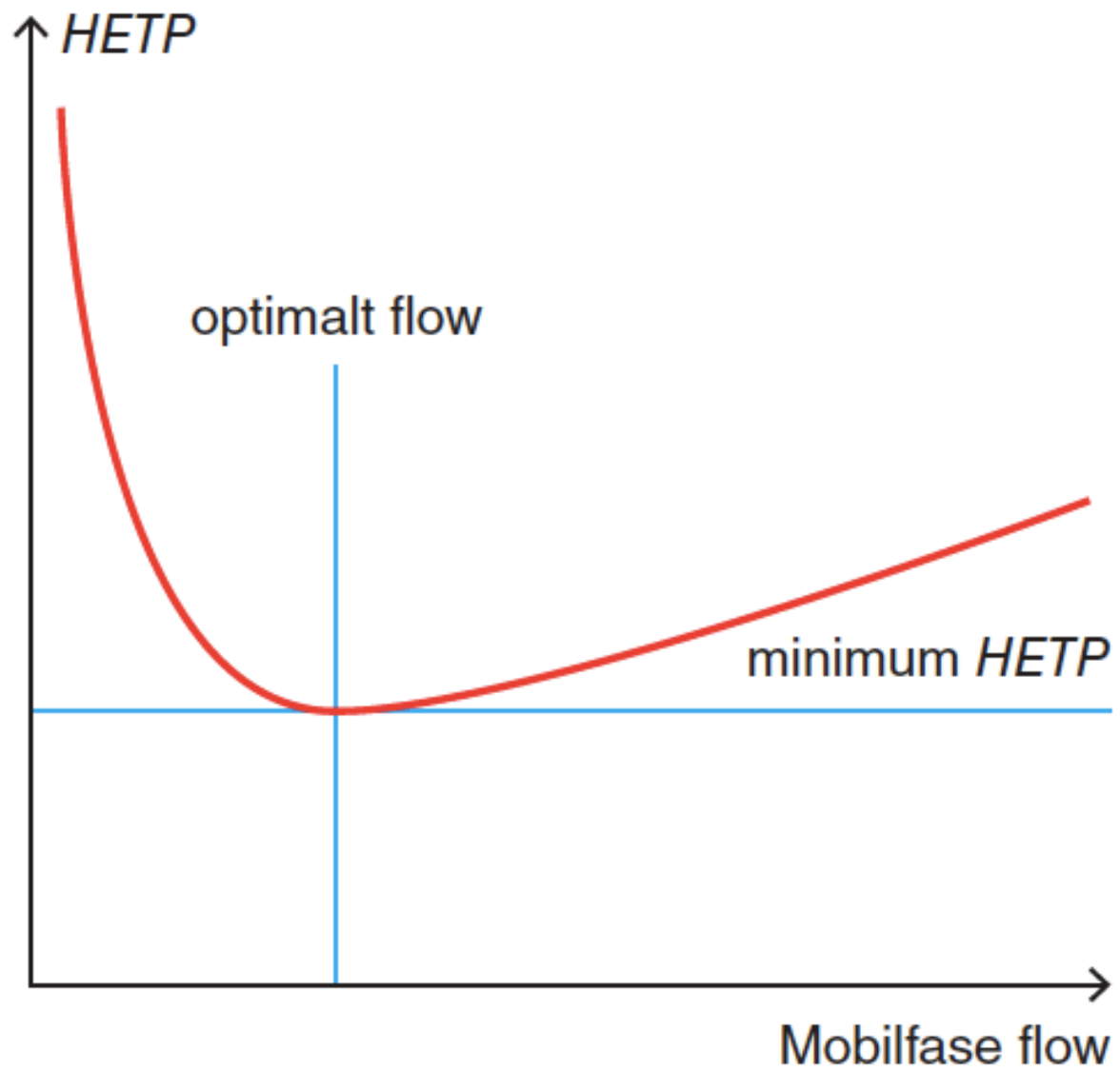
Figur 8.4. Fastlæggelse af retentionstider.



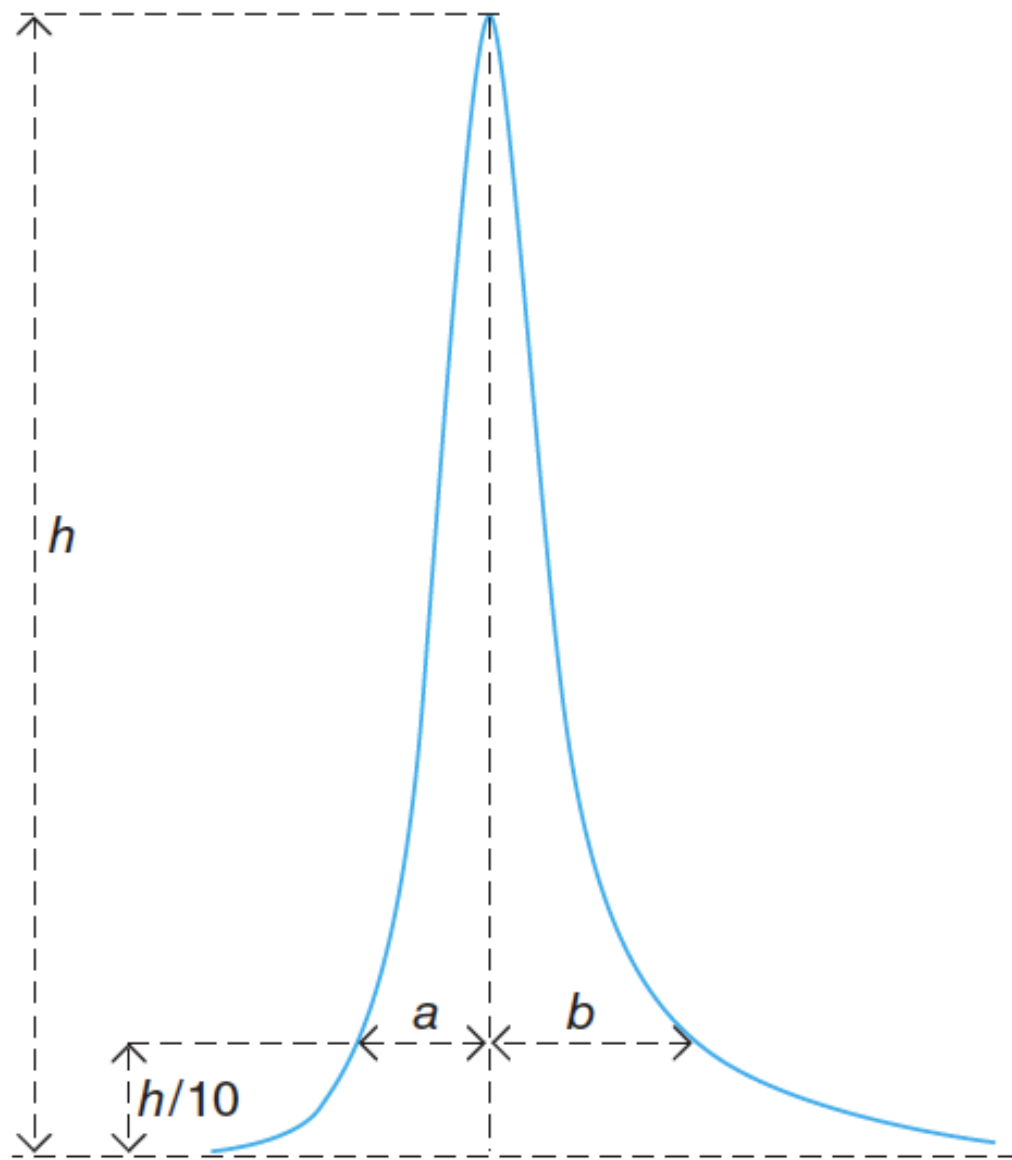
Figur 8.5. Standardchromatogram til identifikationsformål.



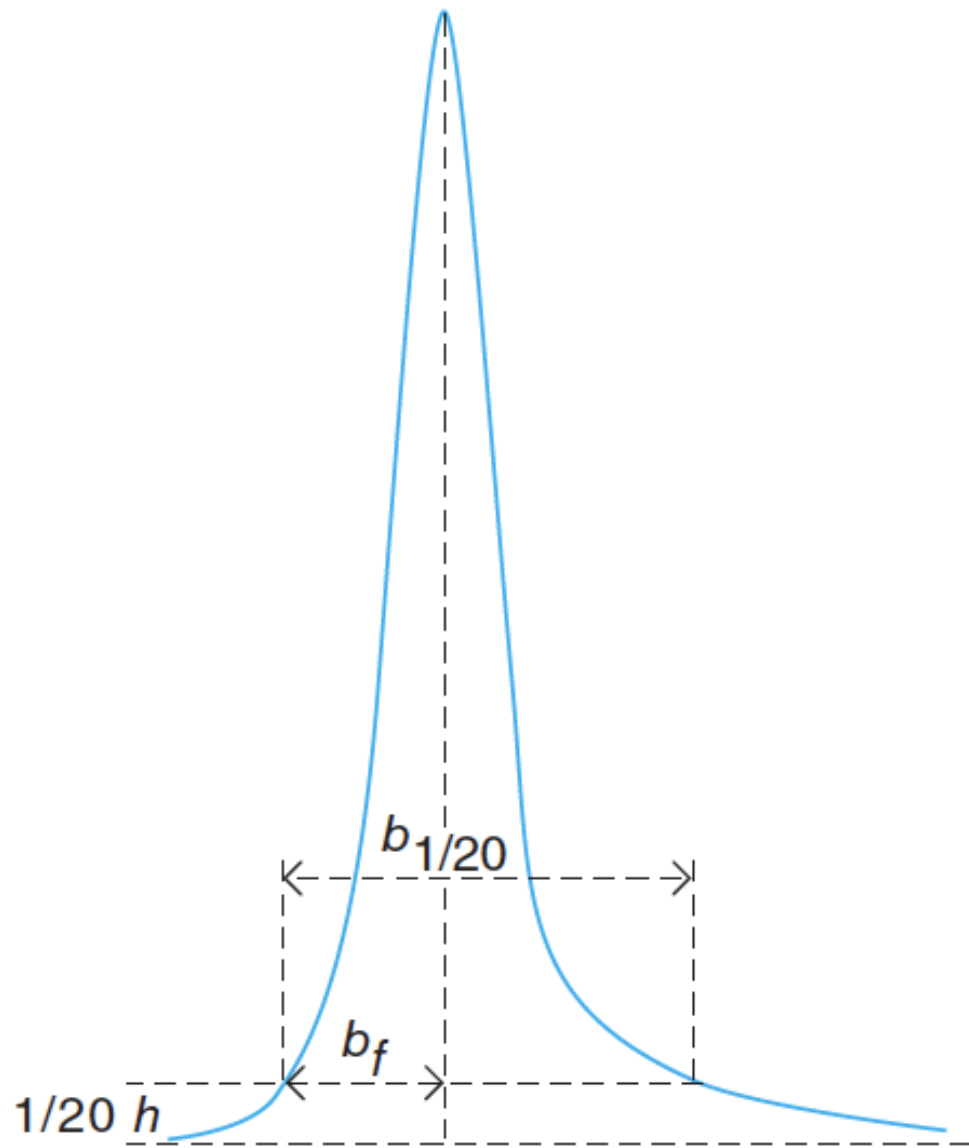
Figur 8.6. Måling af tophøjde og bredden i halv højde.



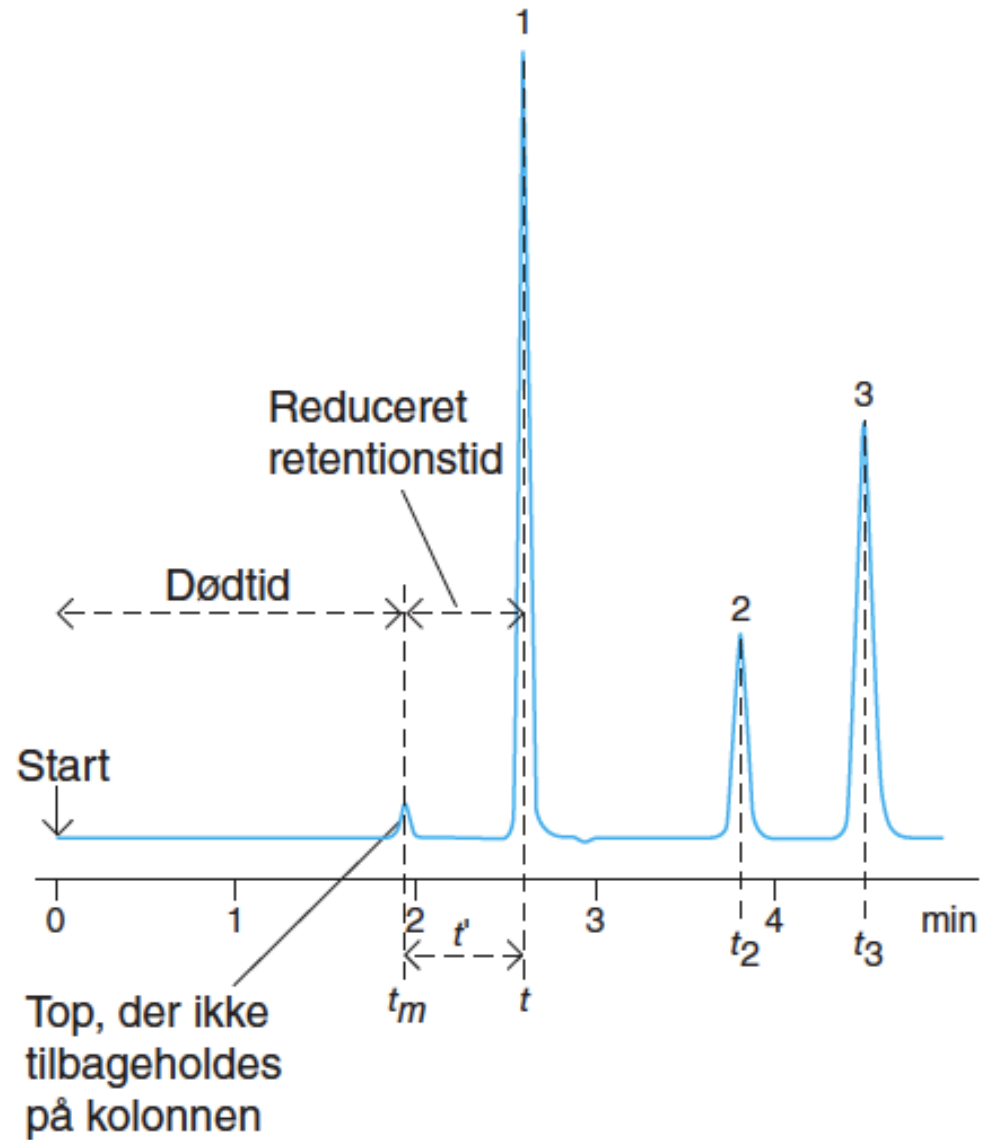
Figur 8.7. van Deemter-kurve.



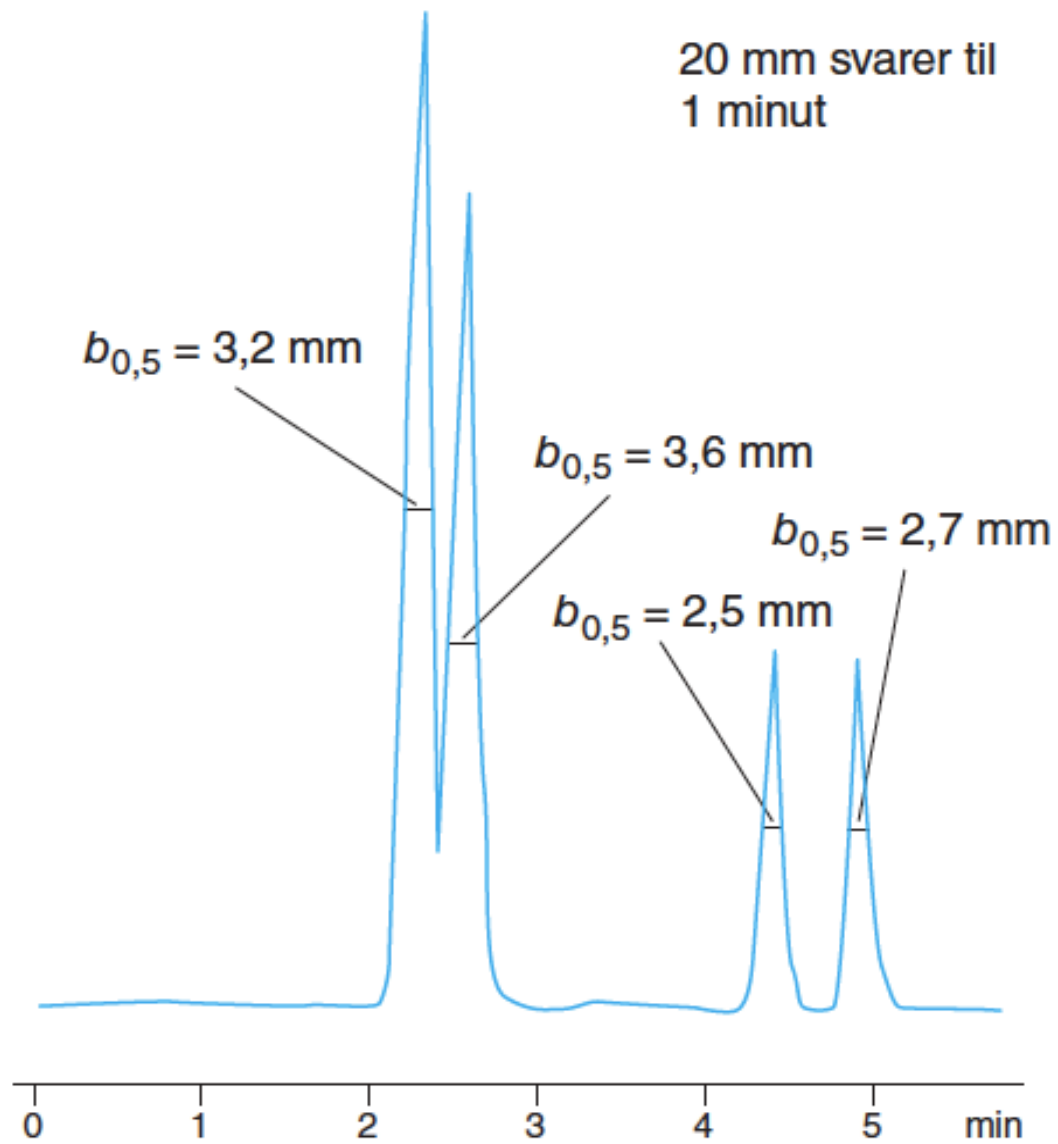
Figur 8.8. Bestemmelse af asymmetrifaktor.



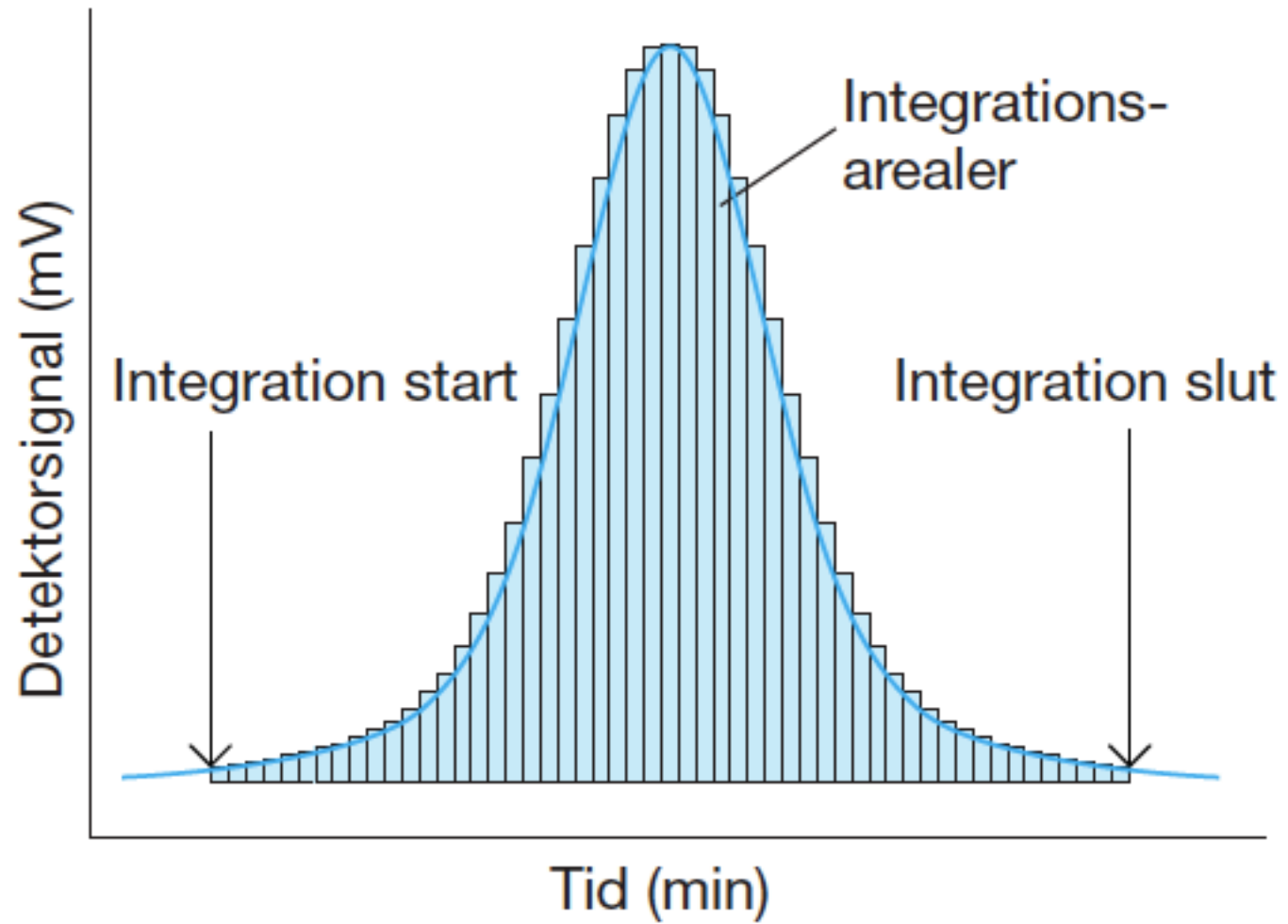
Figur 8.9. Bestemmelse af tailing-faktor.



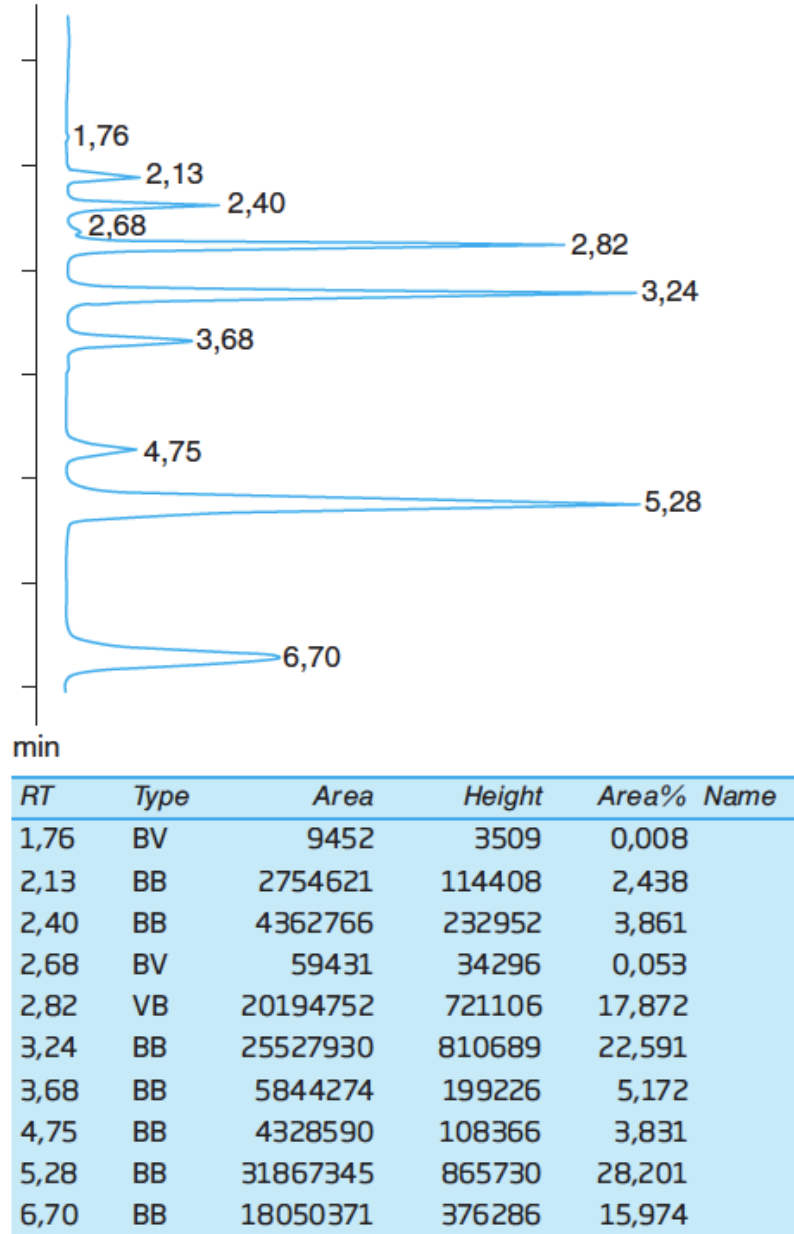
Figur 8.10. Bestemmelse af dødtiden t_m og reduceret retentionstid t' .



Figur 8.11. Bestemmelse af resolutionen R_S .

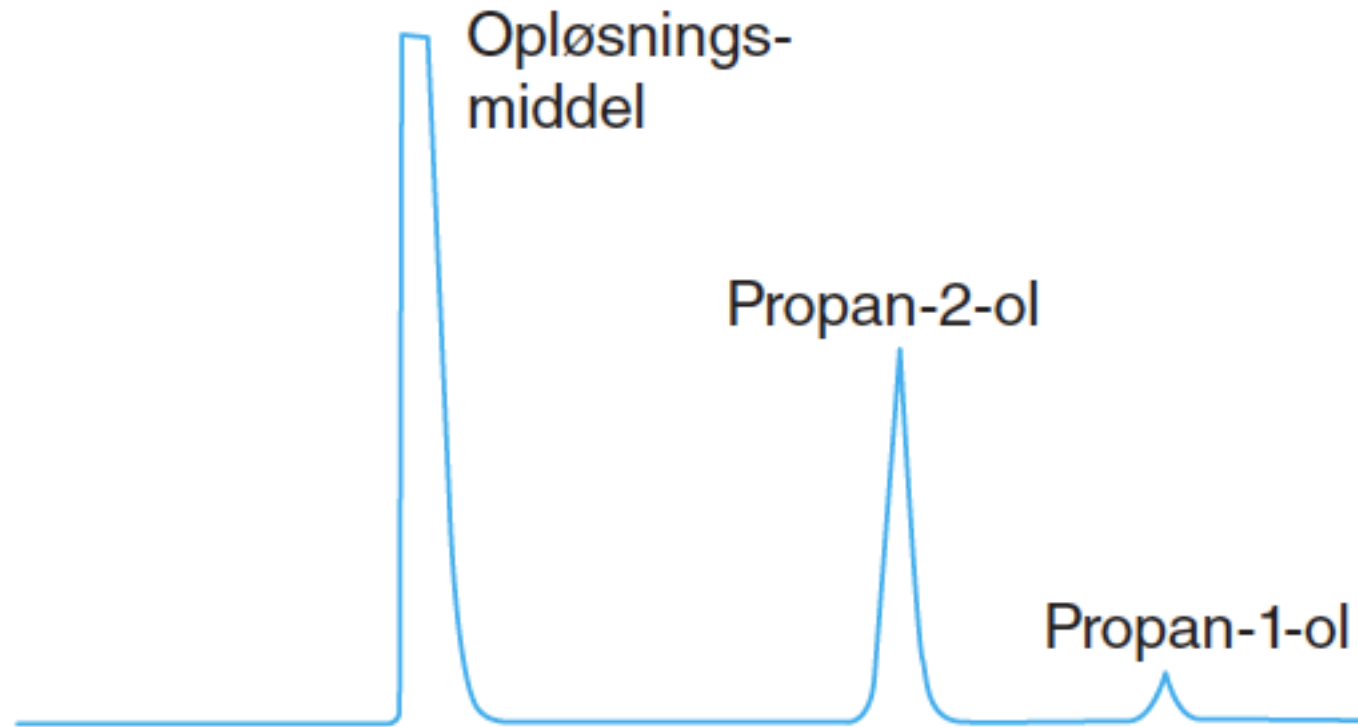


Figur 8.12. Integratorberegning af topareal.



Figur 8.13. Eksempel på integratorudskrift.

Prøveblanding

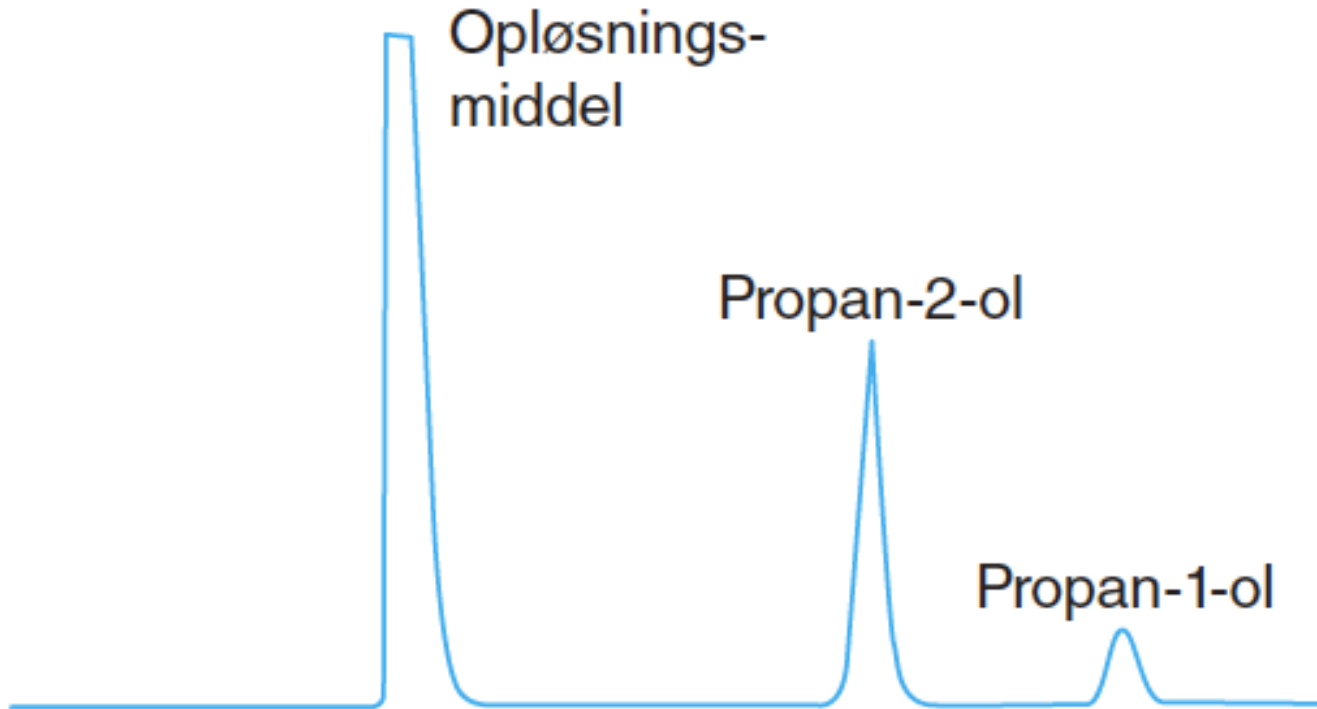


Figur 8.14. Chromatogram af prøven, ekstern standard-metoden.

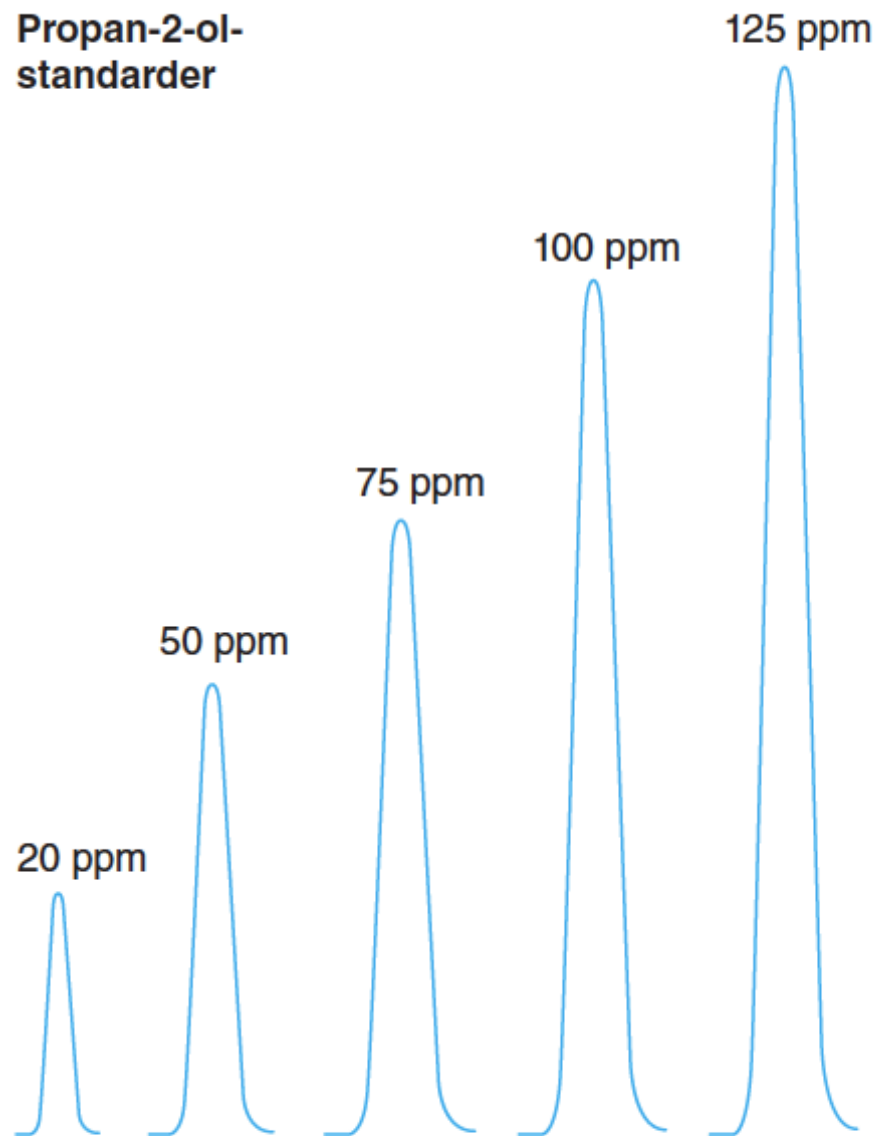
Standardchromatogram

Top A: 50 ppm

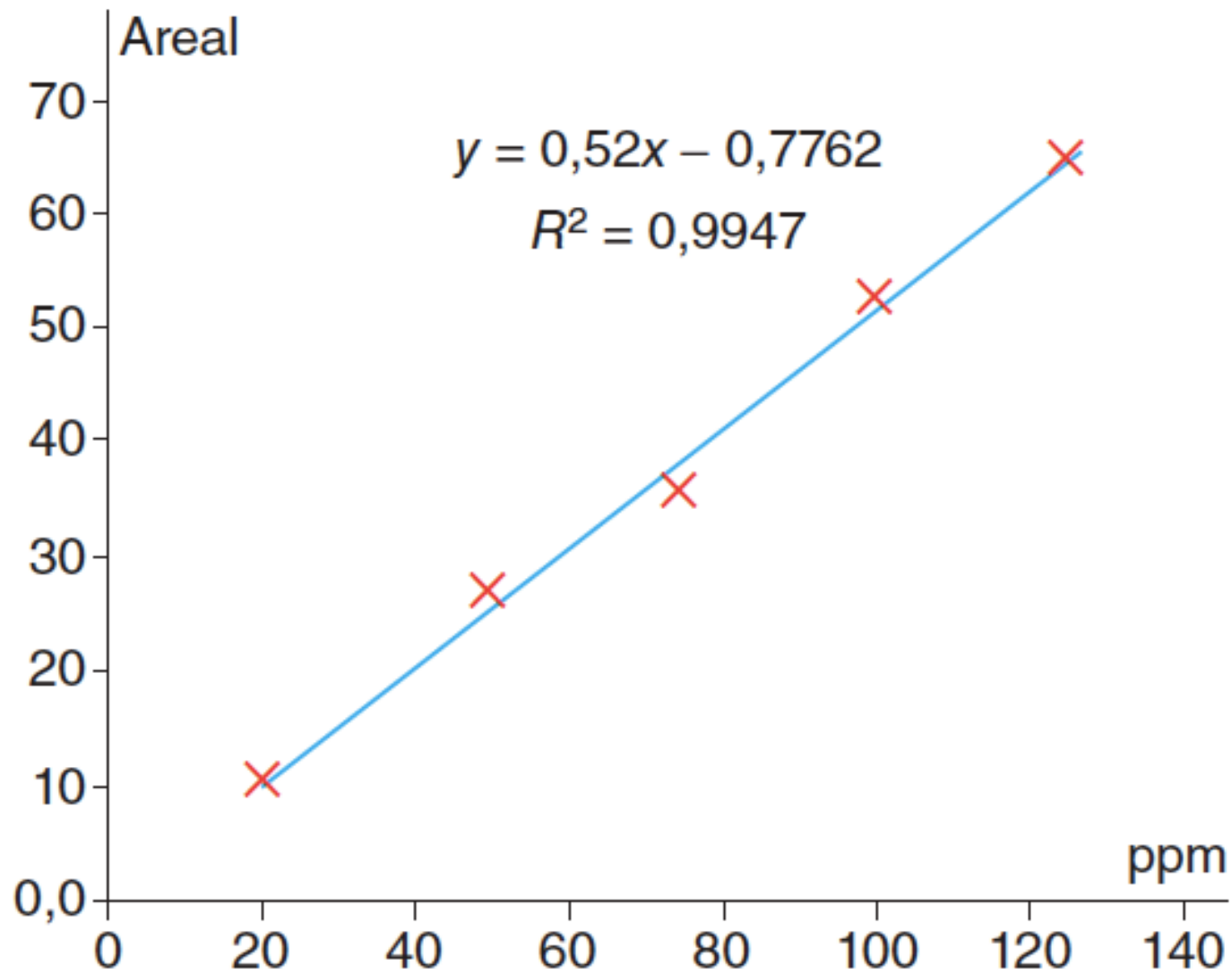
Top B: 10 ppm



Figur 8.15. Chromatogram af standarderne, ekstern standard-metoden.

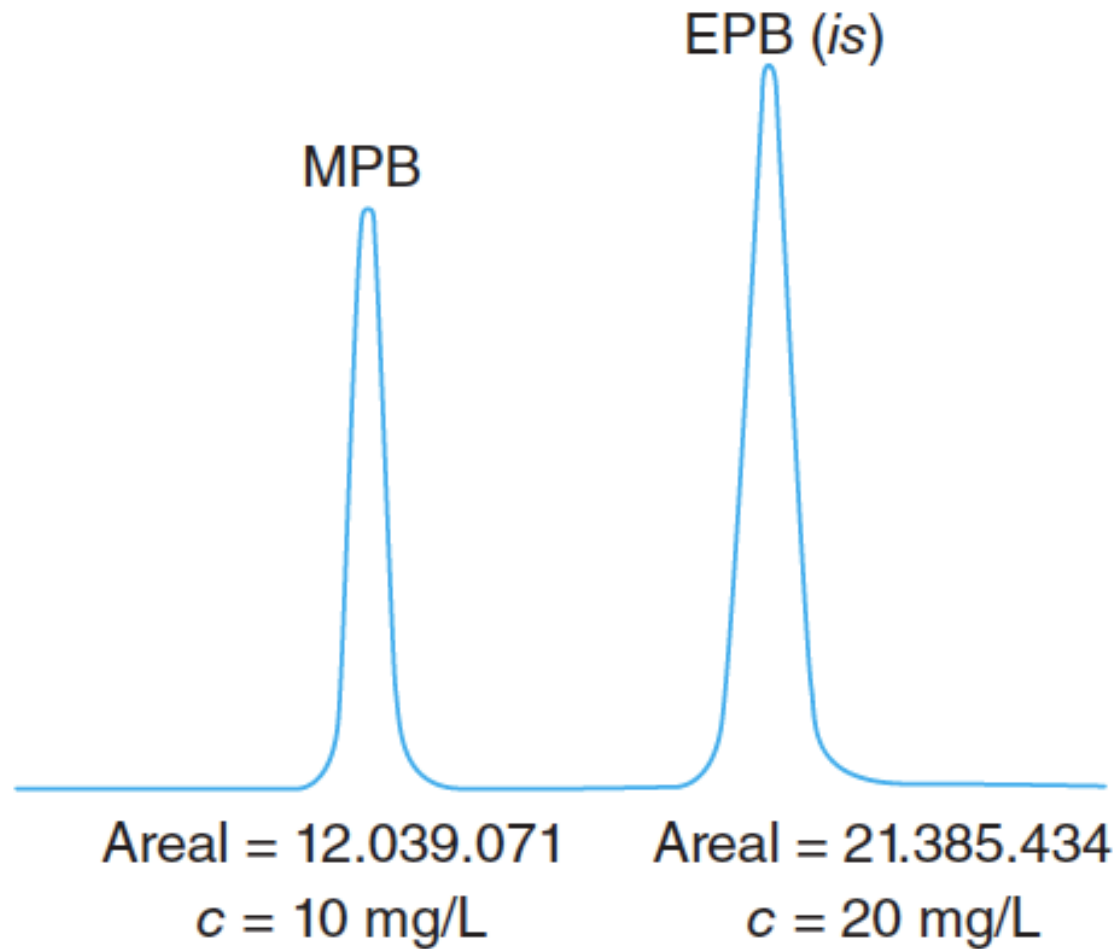


Figur 8.16. Koncentrationsbestemmelse med standardkurve.



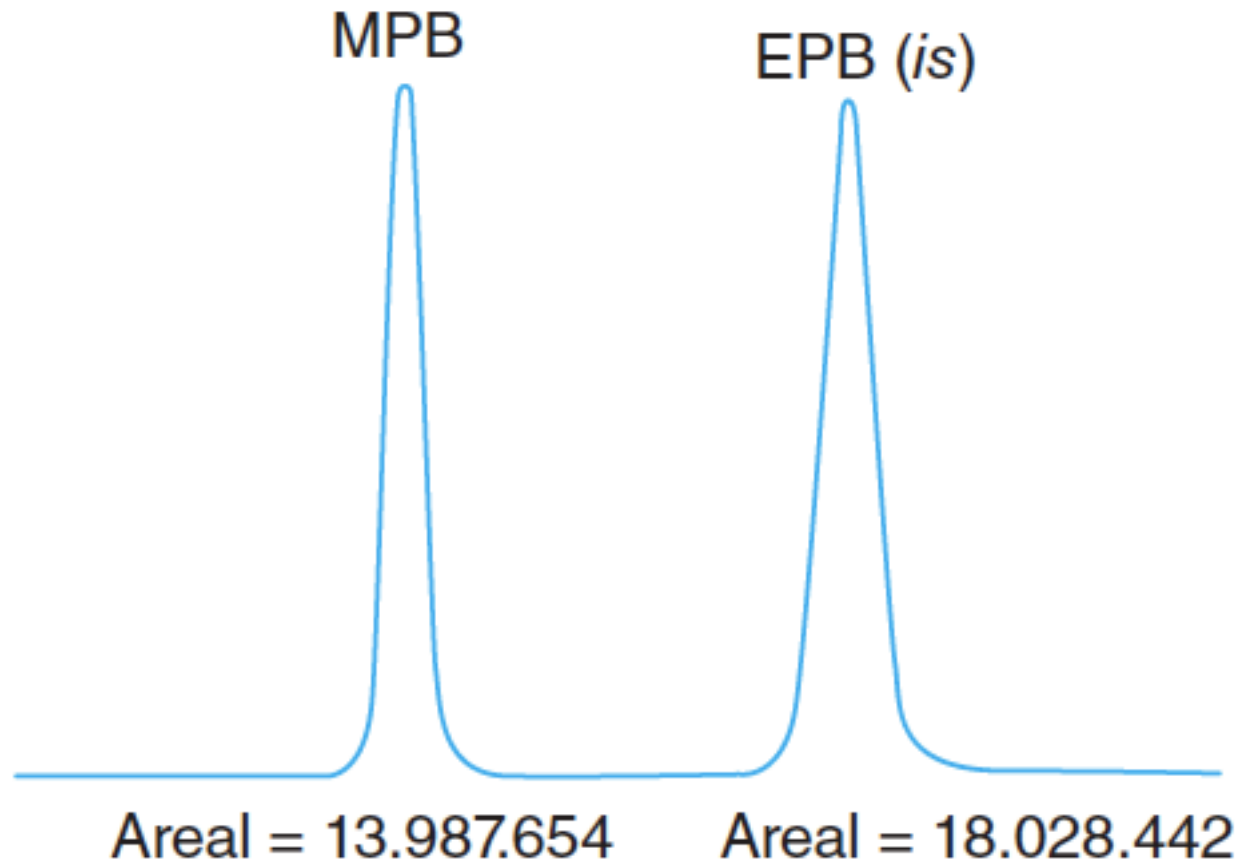
Figur 8.17. Standardkurve.

Standardblanding

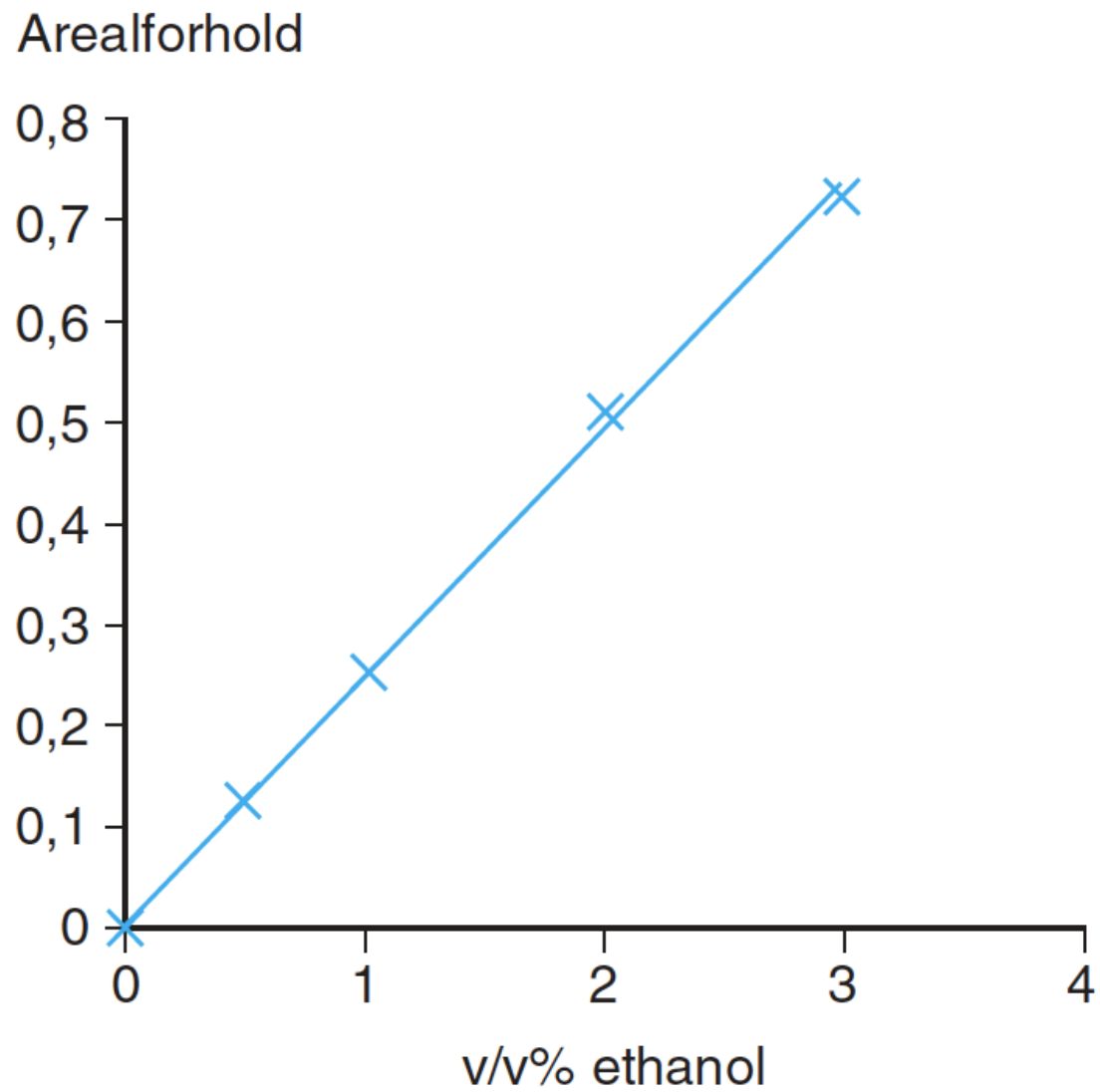


Figur 8.18. Standardchromatogram til bestemmelse af f_{rel} , intern standard-metoden.

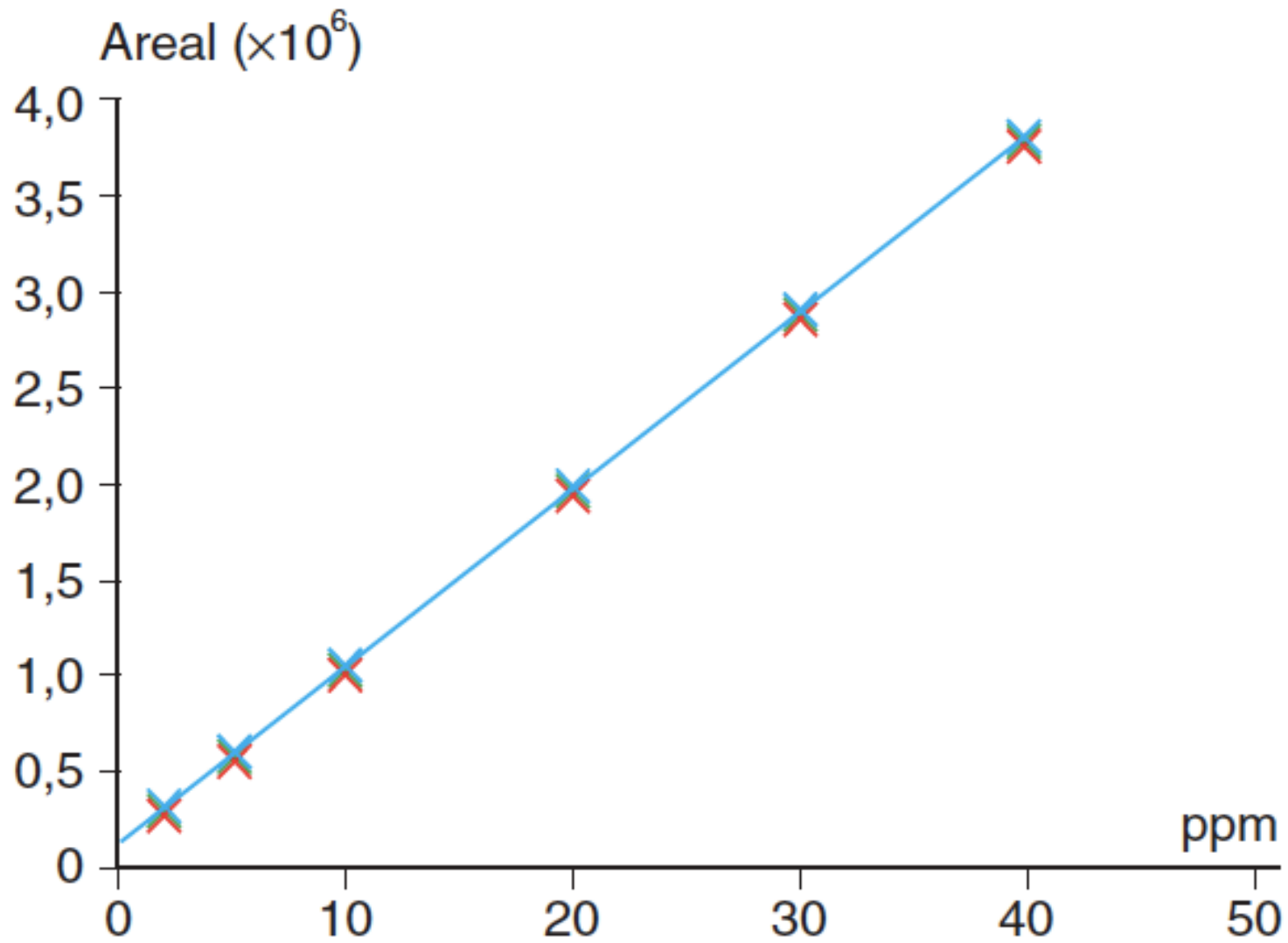
Prøve med tilsat intern standard



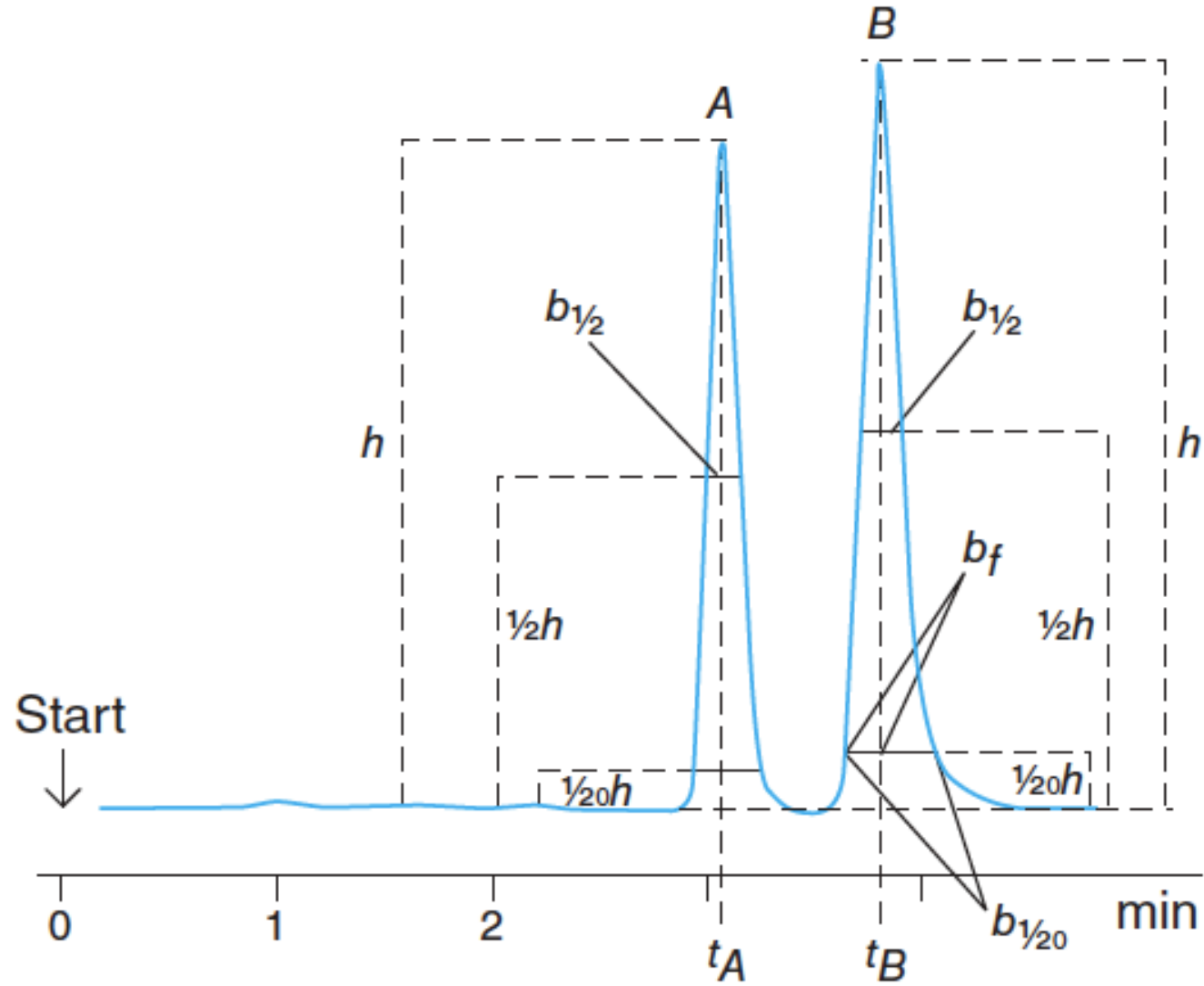
Figur 8.19. Chromatogram til bestemmelse af prøvekoncentration, intern standard-metoden.



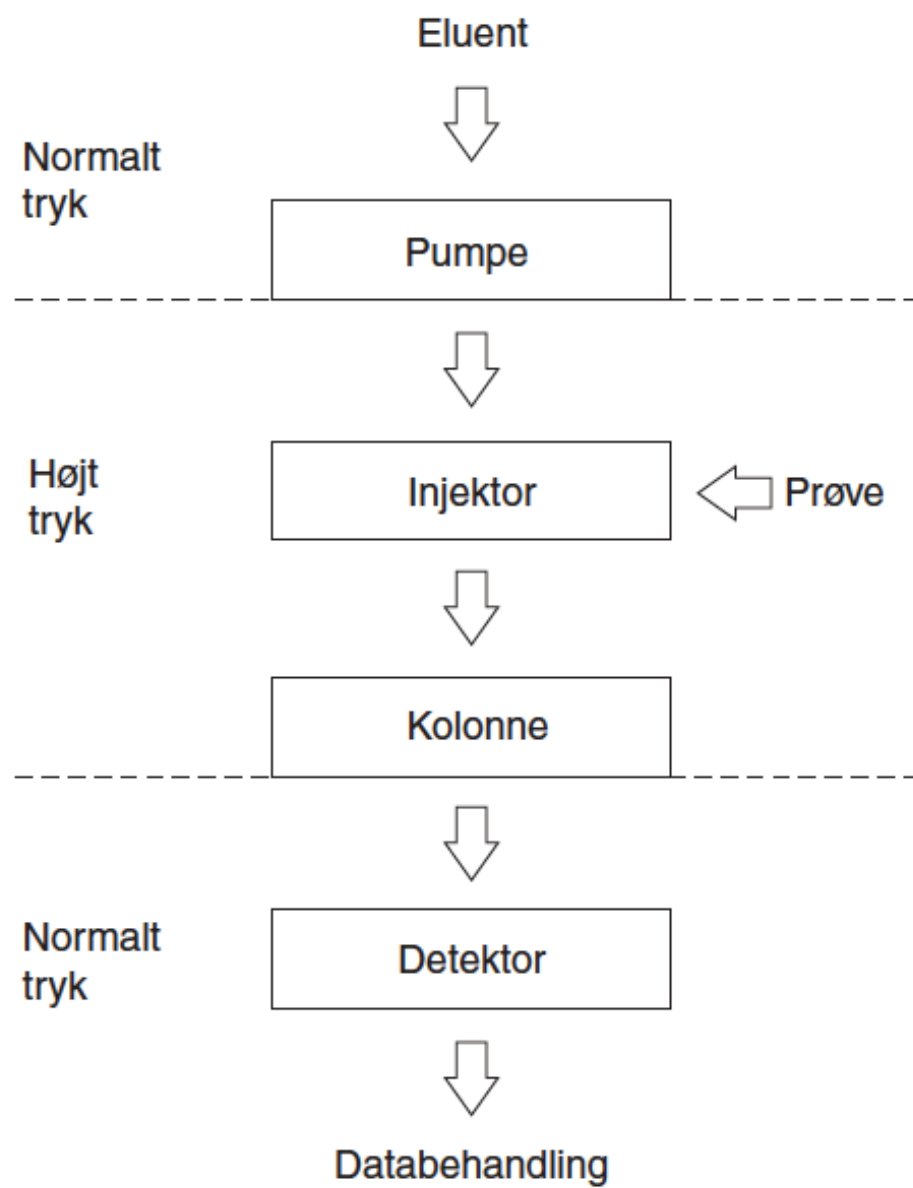
Figur 8.20. Standardkurve for ethanol tilsat intern standard.



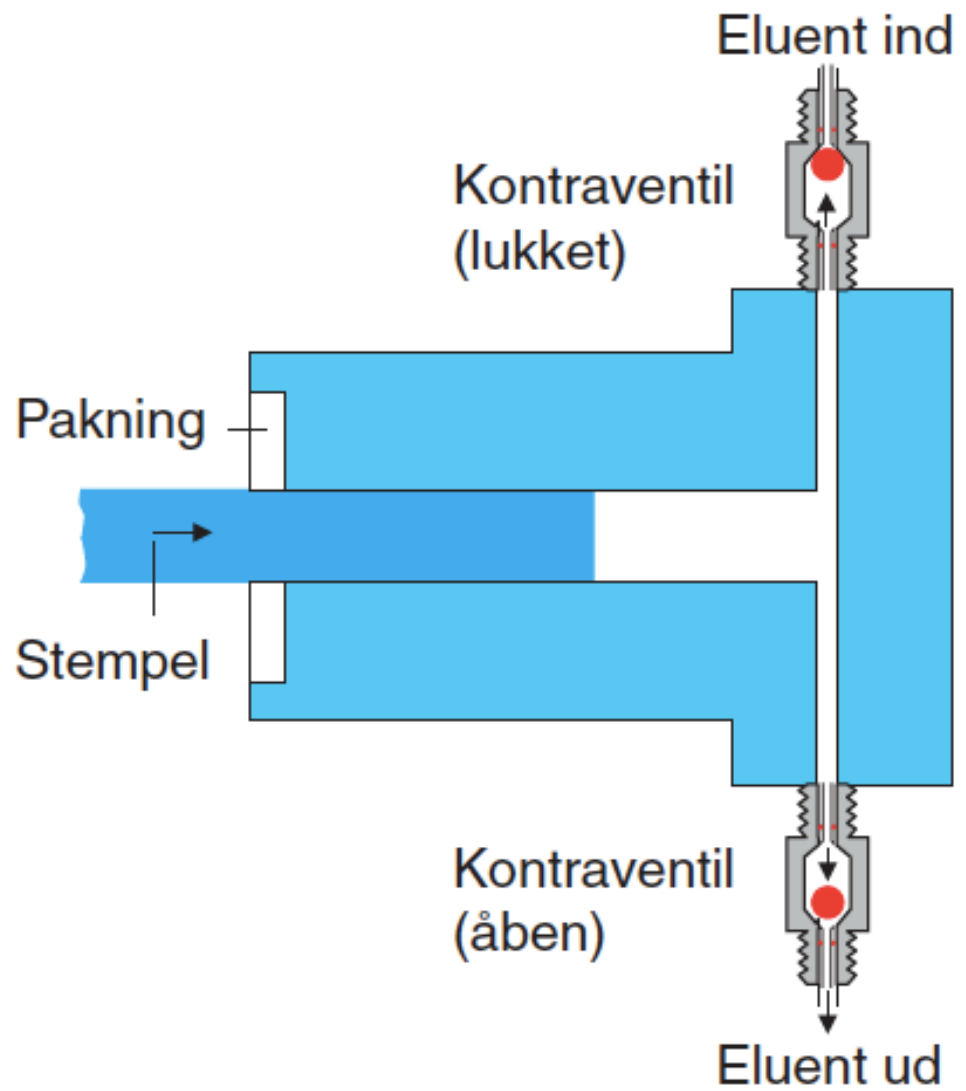
Figur 8.21. Linearitetsbestemmelse.



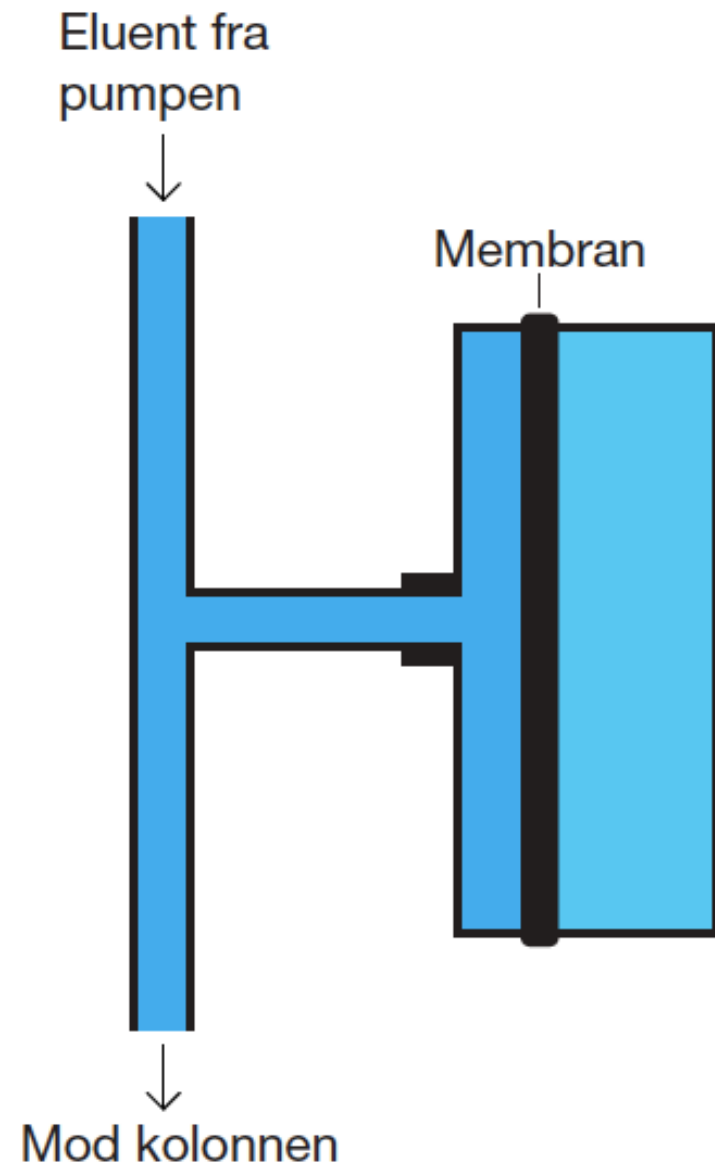
Figur 8.22. Chromatogram til bestemmelse af selektivitetsparametrene N , R_s og $T_{5\%}$.



Figur 9.1. HPLC-systemets opbygning.



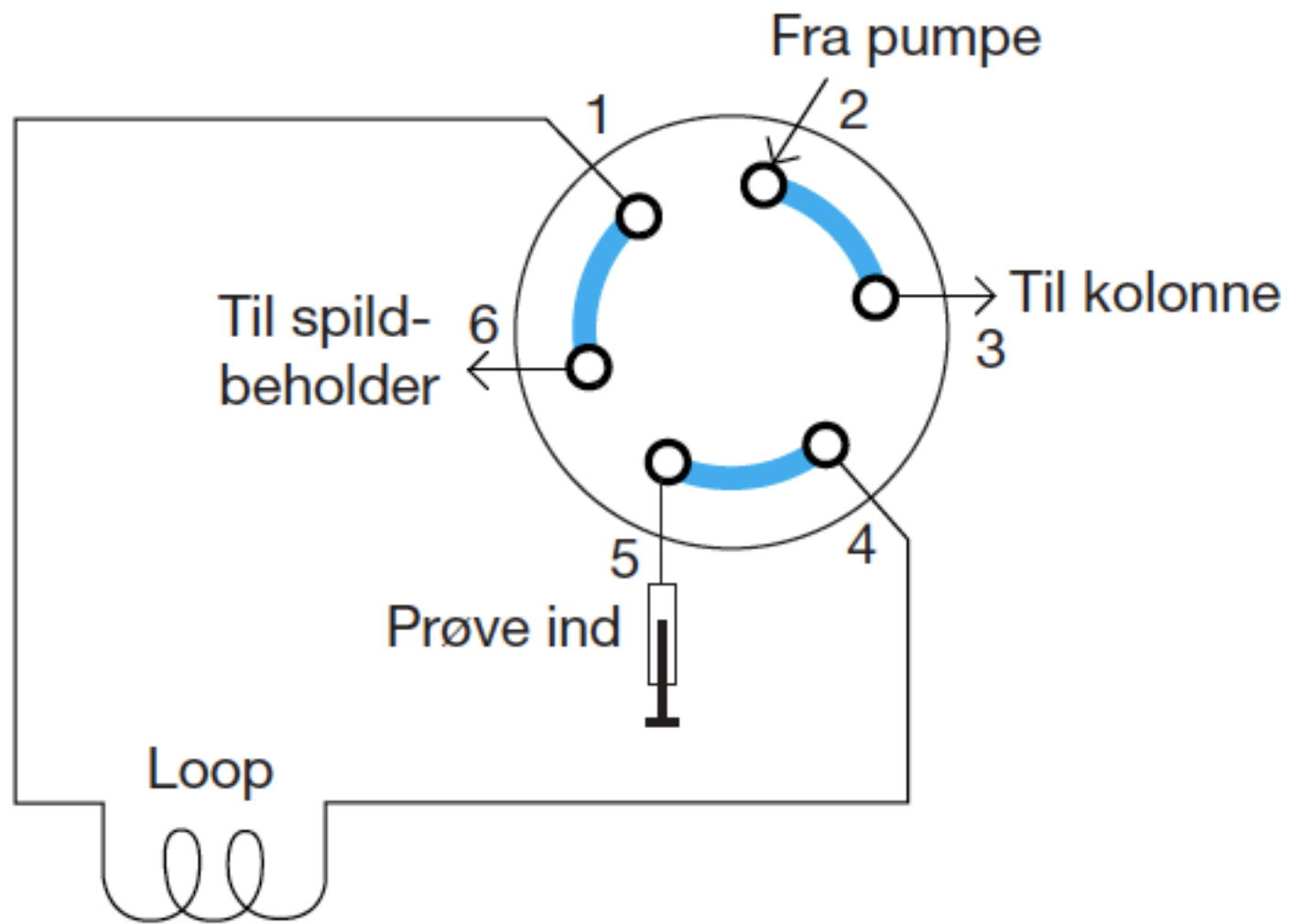
Figur 9.2. HPLC-pumpens virkemåde (tømning).



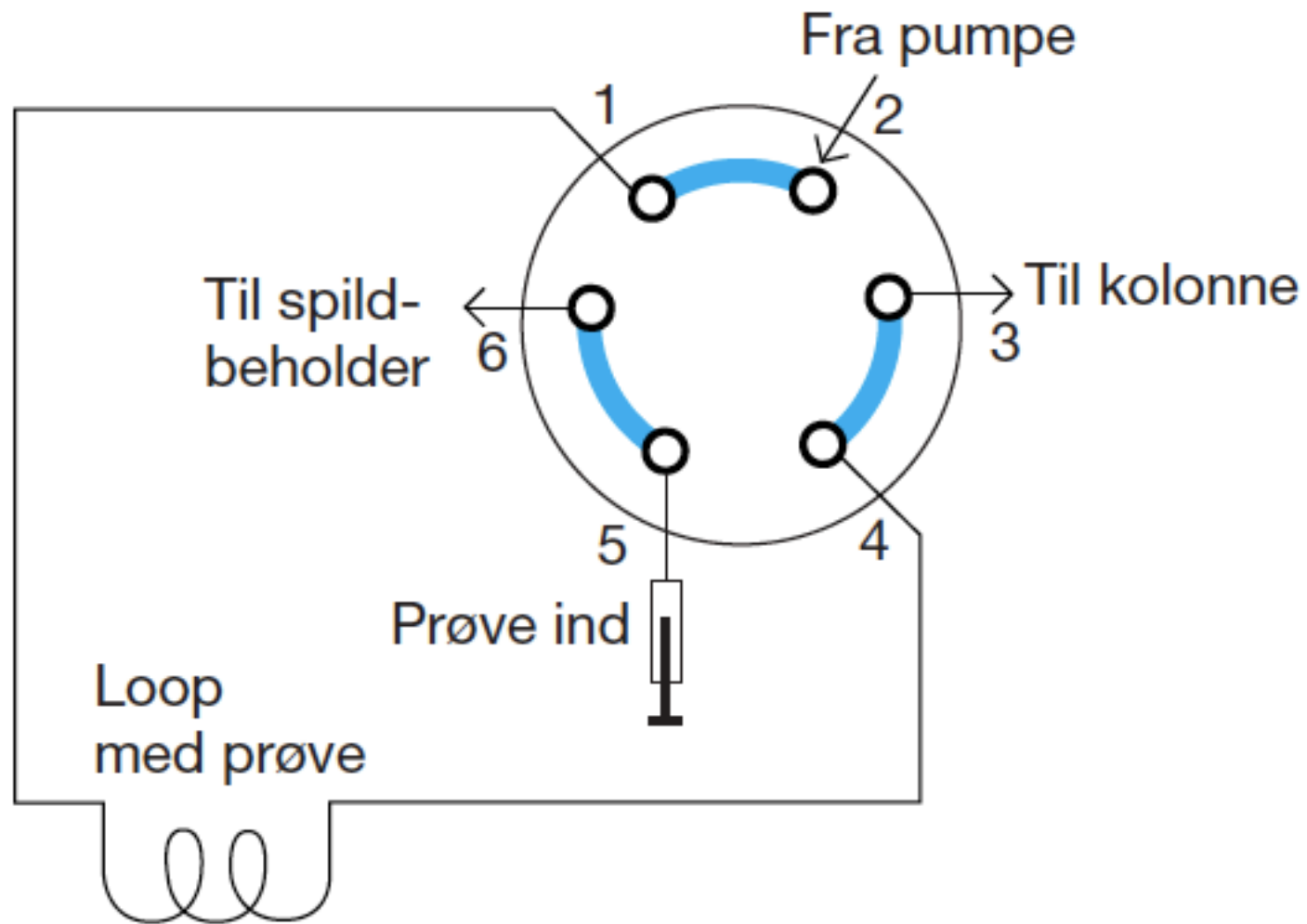
Figur 9.3. Pulsdæmperen.



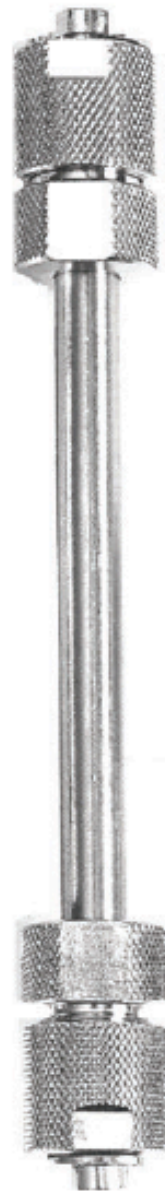
Figur 9.4. Rheodyne injektionsventil.



Figur 9.5. Injektionsventilen i position ``Load''.



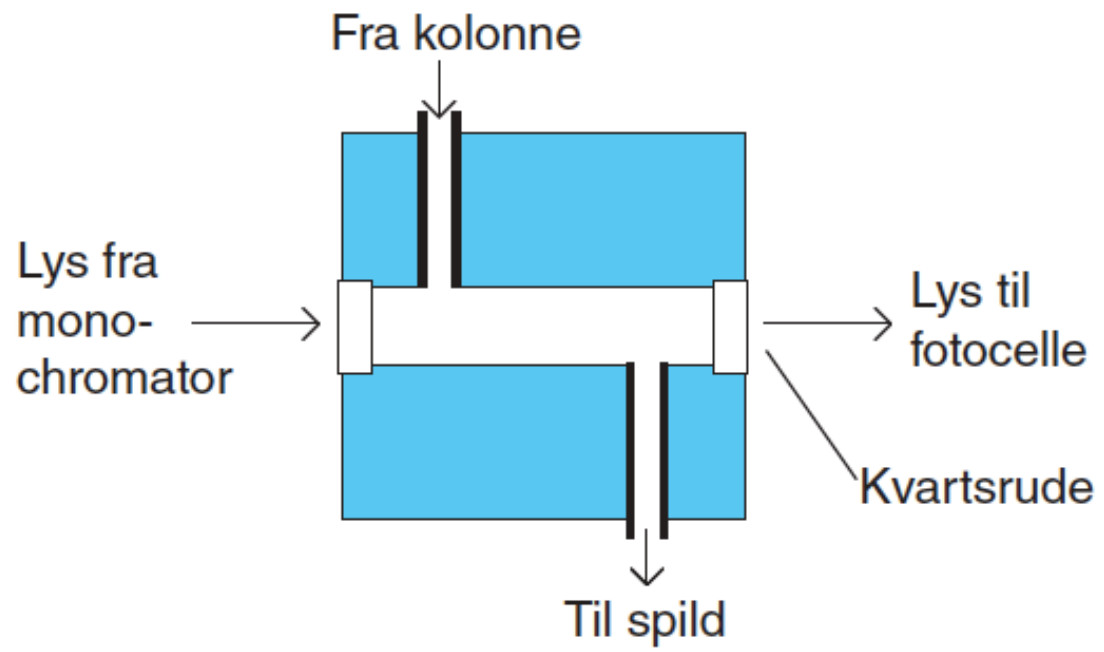
Figur 9.6. Injektionsventilen i position "Inject".



Figur 9.7. HPLC-kolonne.

<i>Type</i>	<i>Kvantifikationsgrænse (ng)</i>	<i>Linaritetsgrænse (ng)</i>
UV	0,1	1000
DAD	1	2000
Fluorescens	0,001	0,1
Refraktionsindeks	10	10000
Ledningsevne	0,1	1000
Elektrokemisk	0,01	10
MS	0,05	-
ELS	20	10000

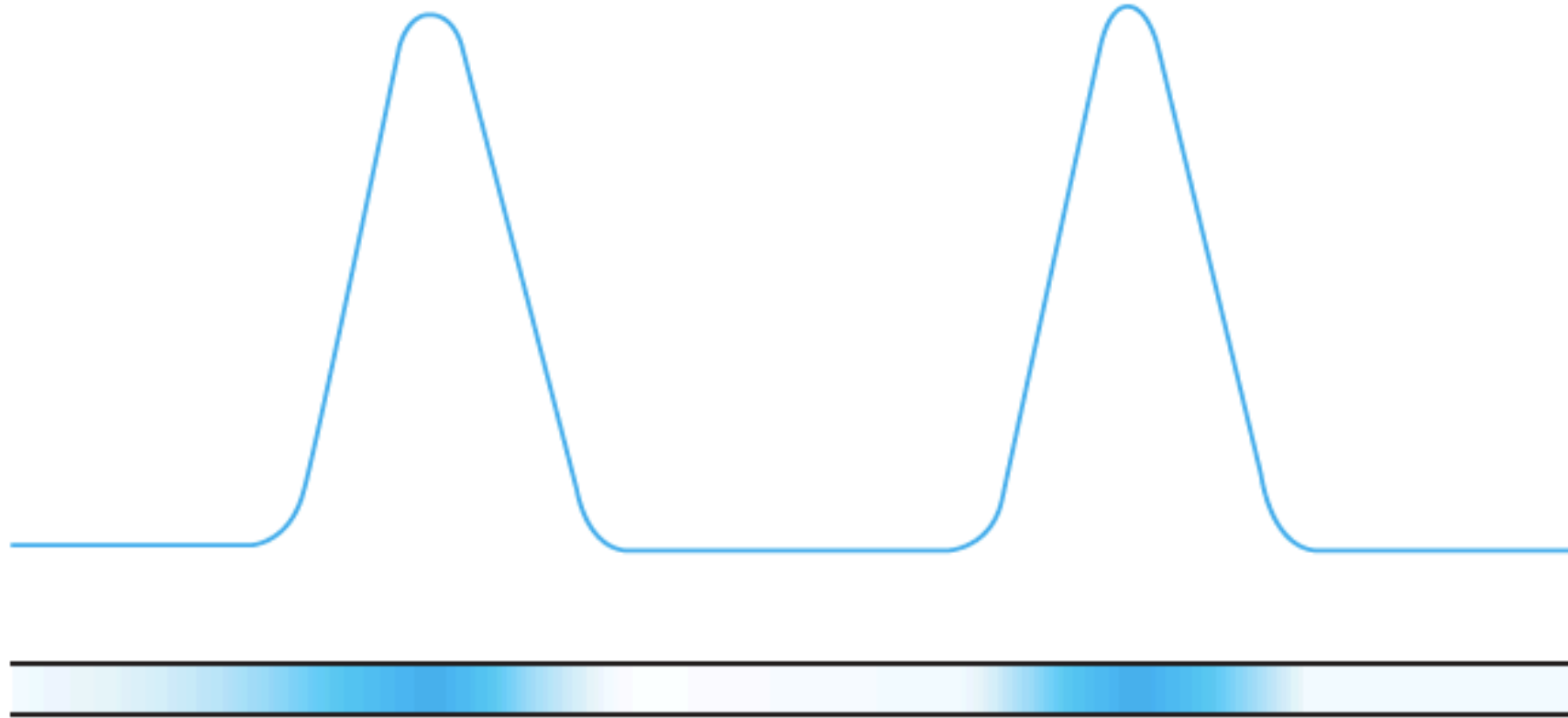
Tabel 9.1. Værdierne er kun omtrentlige niveauer, for grænserne er meget afhængige af, hvilket stof der måles på, samt hvilket udstyr der anvendes. Niveauerne er venligst oplyst af Tore Vulpius, MSC ApS.



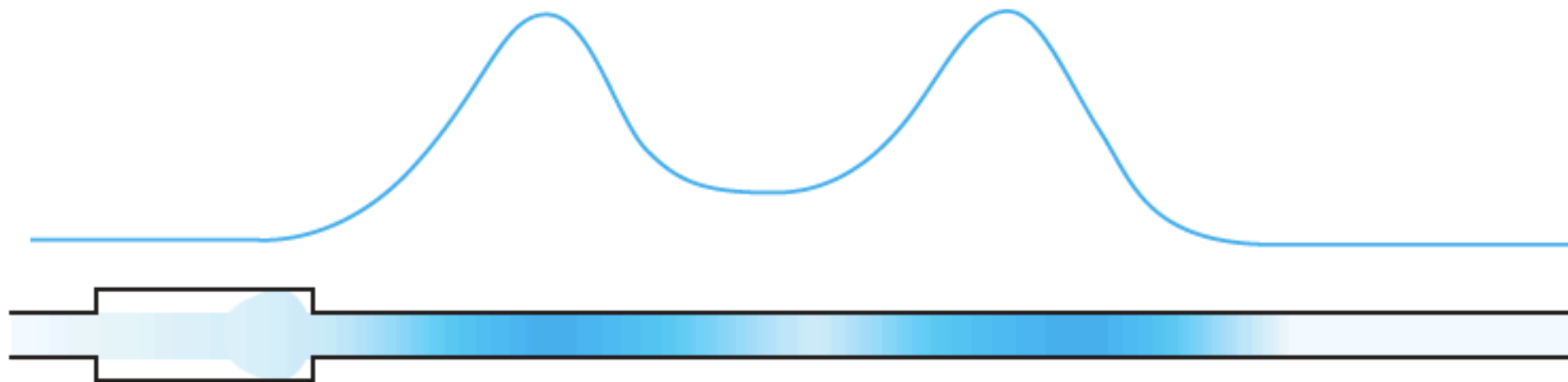
Figur 9.8. Flowcelle til UV-detektor.



Figur 9.9. Adskillelse af komponenterne i kolonnen.

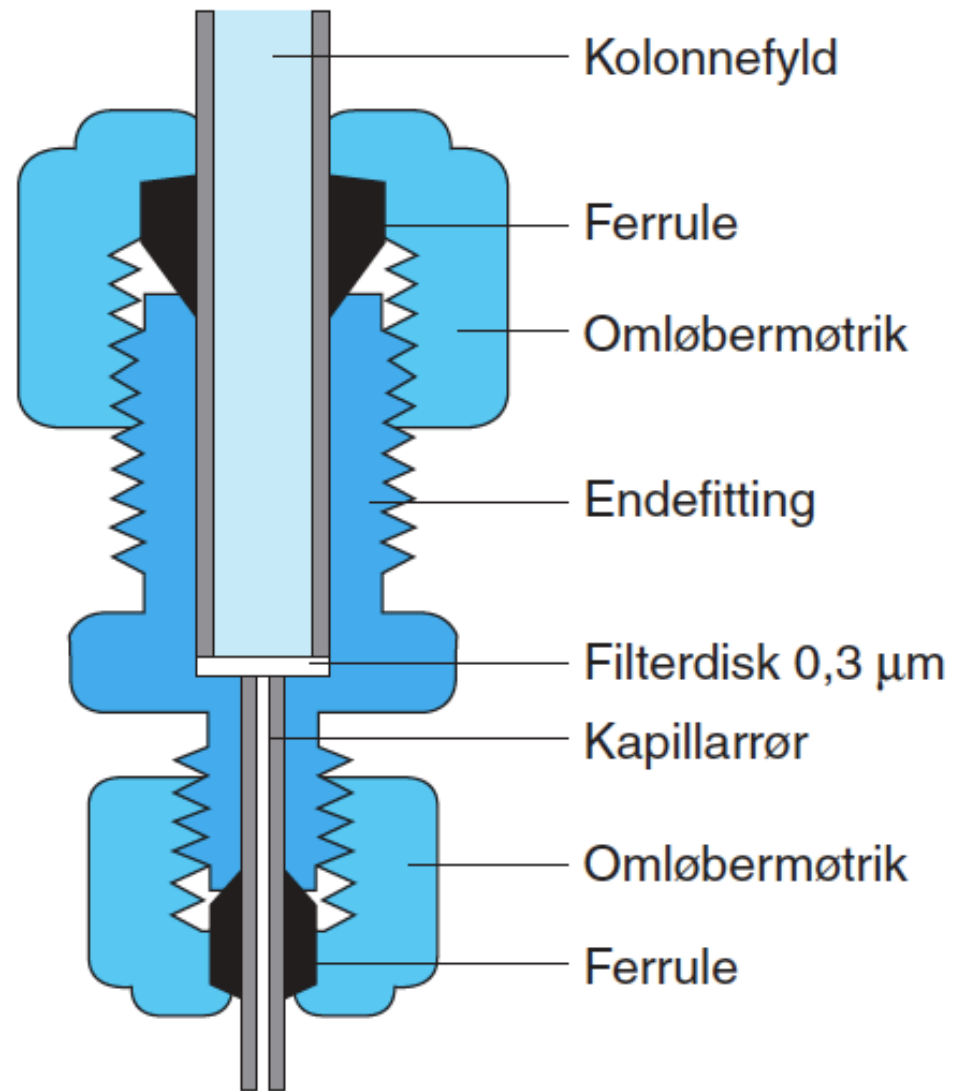


Figur 9.10. Registrering af toppe uden dødvolumen i systemet.

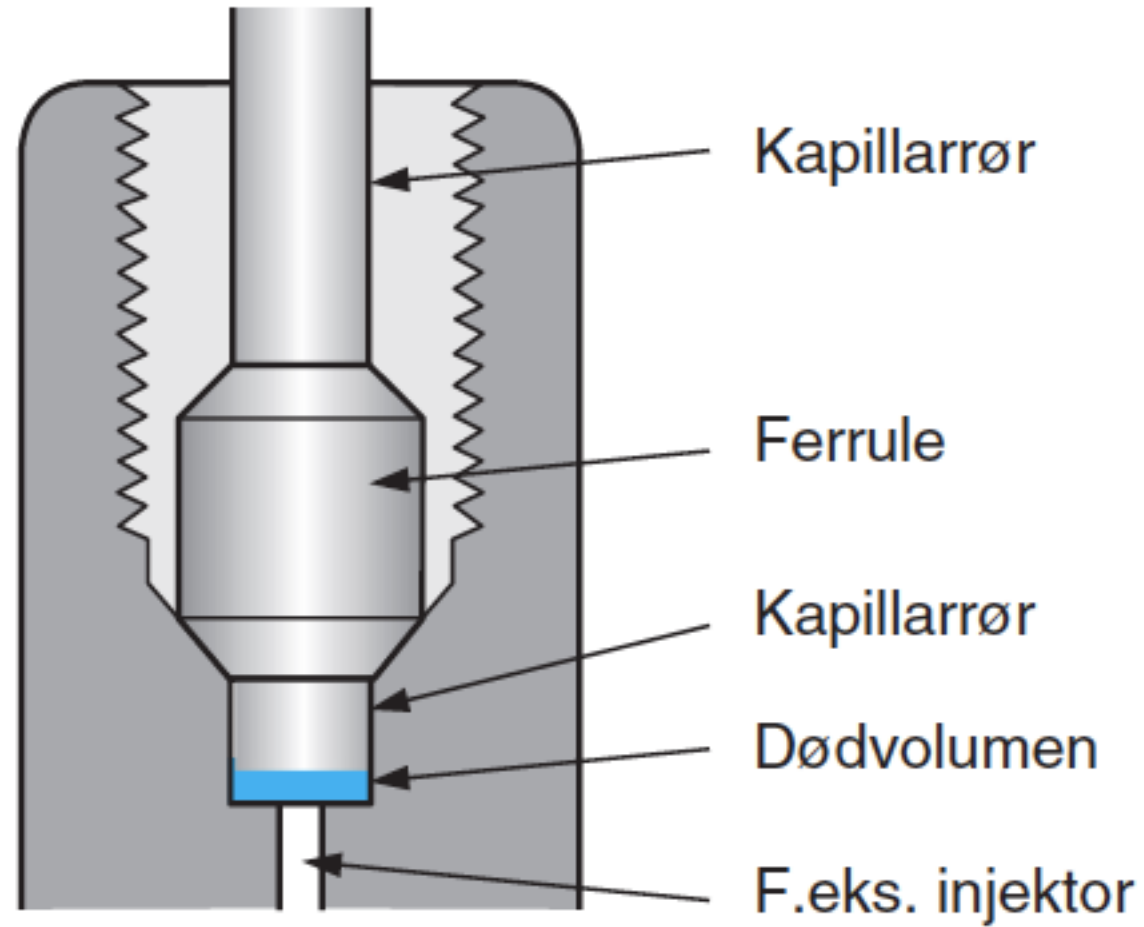


Dødvolumen

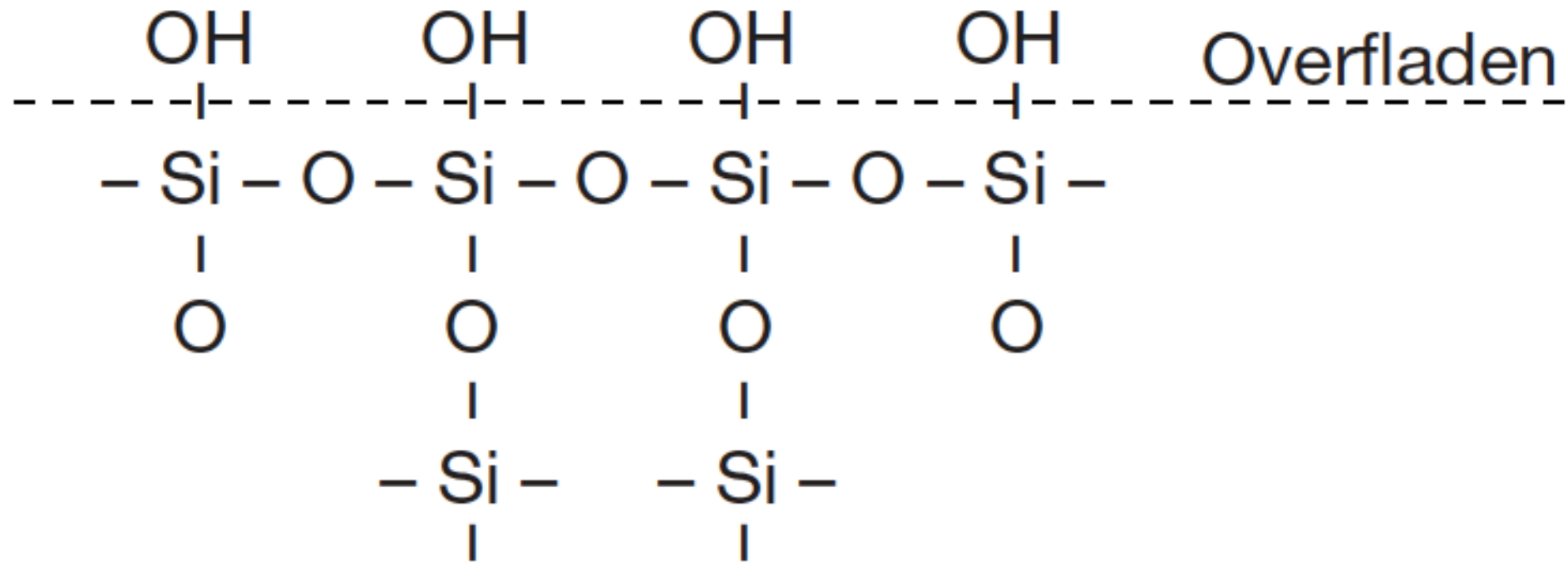
Figur 9.11. Registrering af toppe med dødvolumen i systemet.



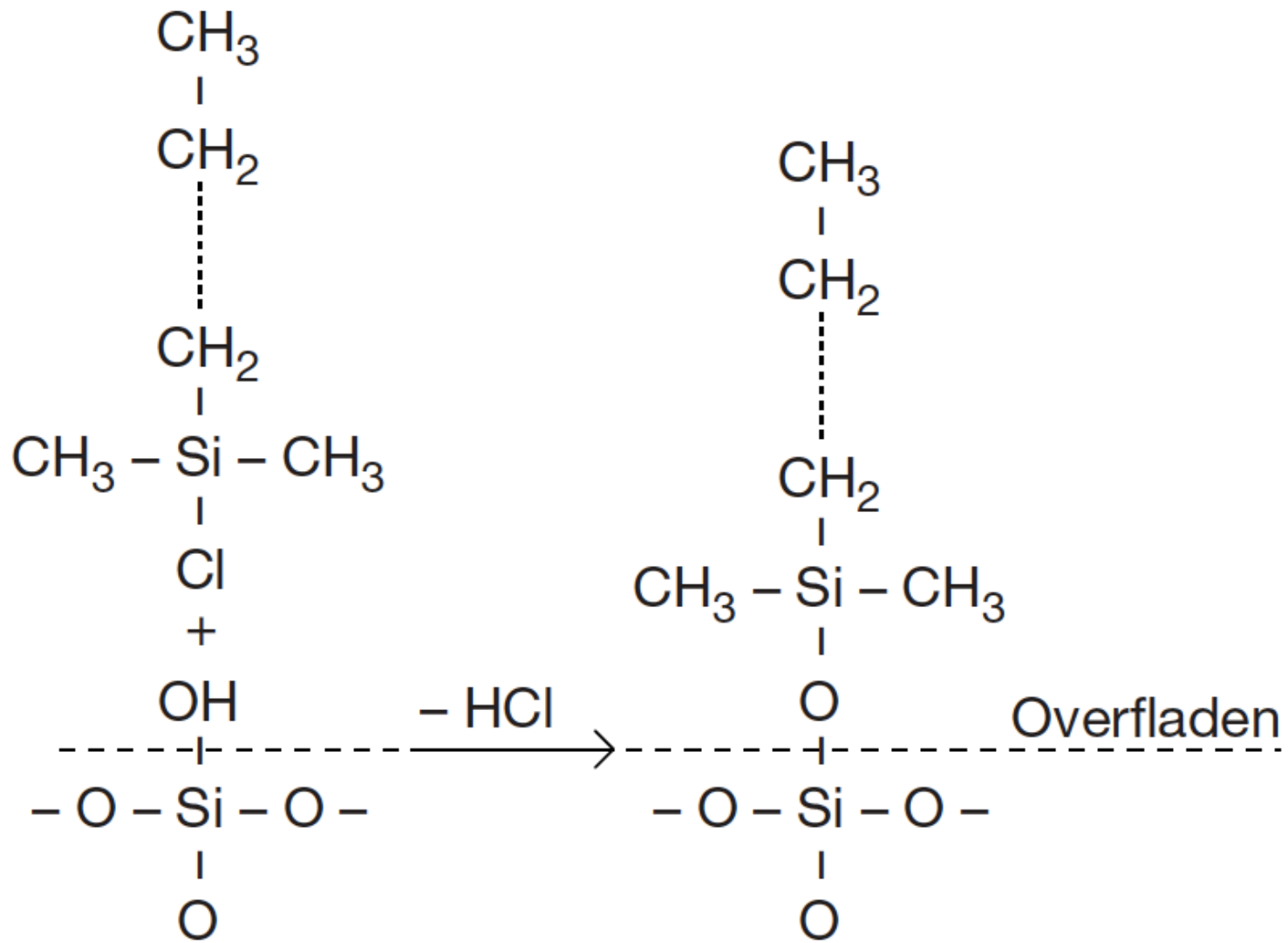
Figur 9.12. Samling af kolonne og kapillarrør.



Figur 9.13. Montering af kapillarrør



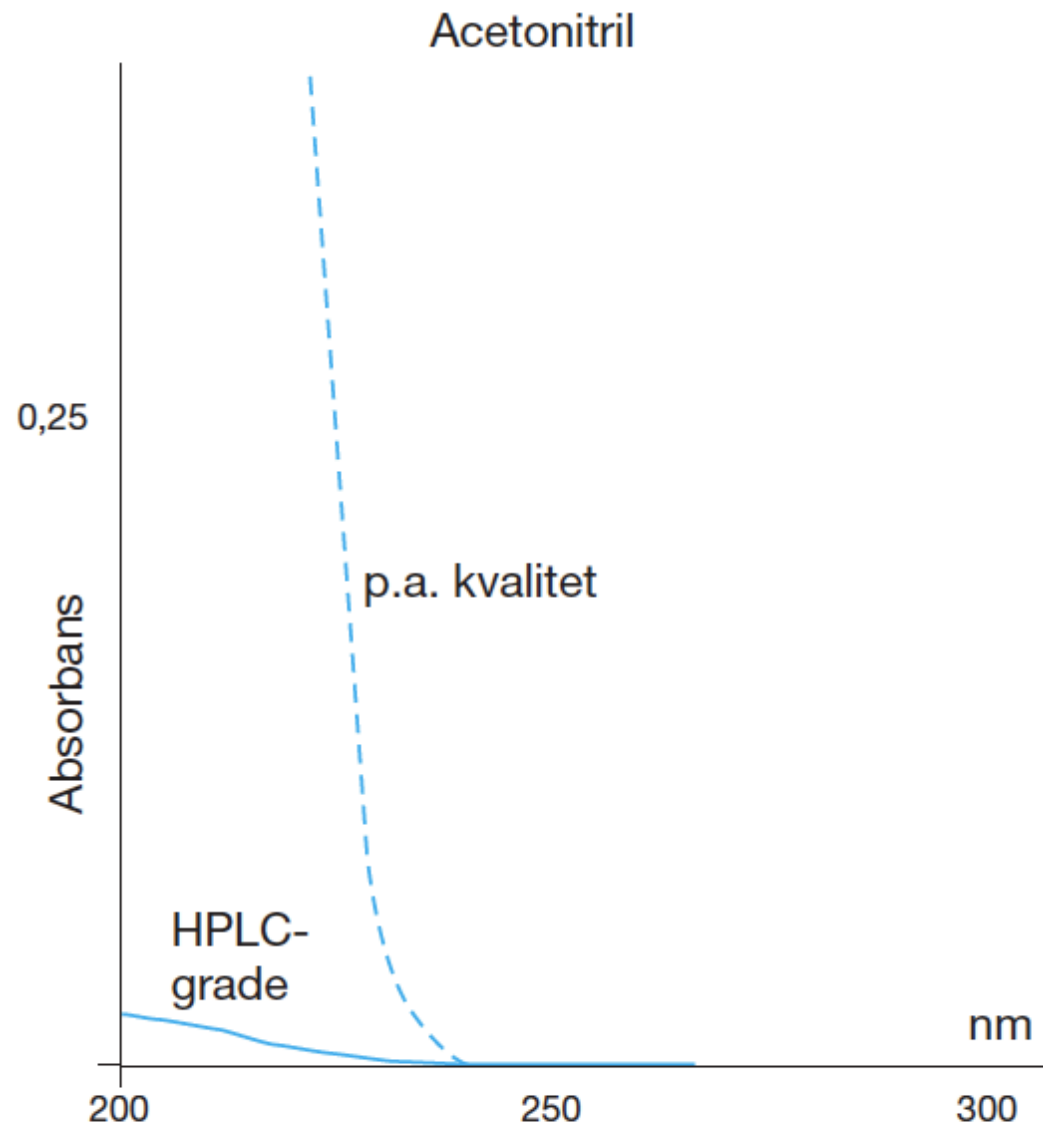
Figur 9.14. Silikatkornenes overflade.



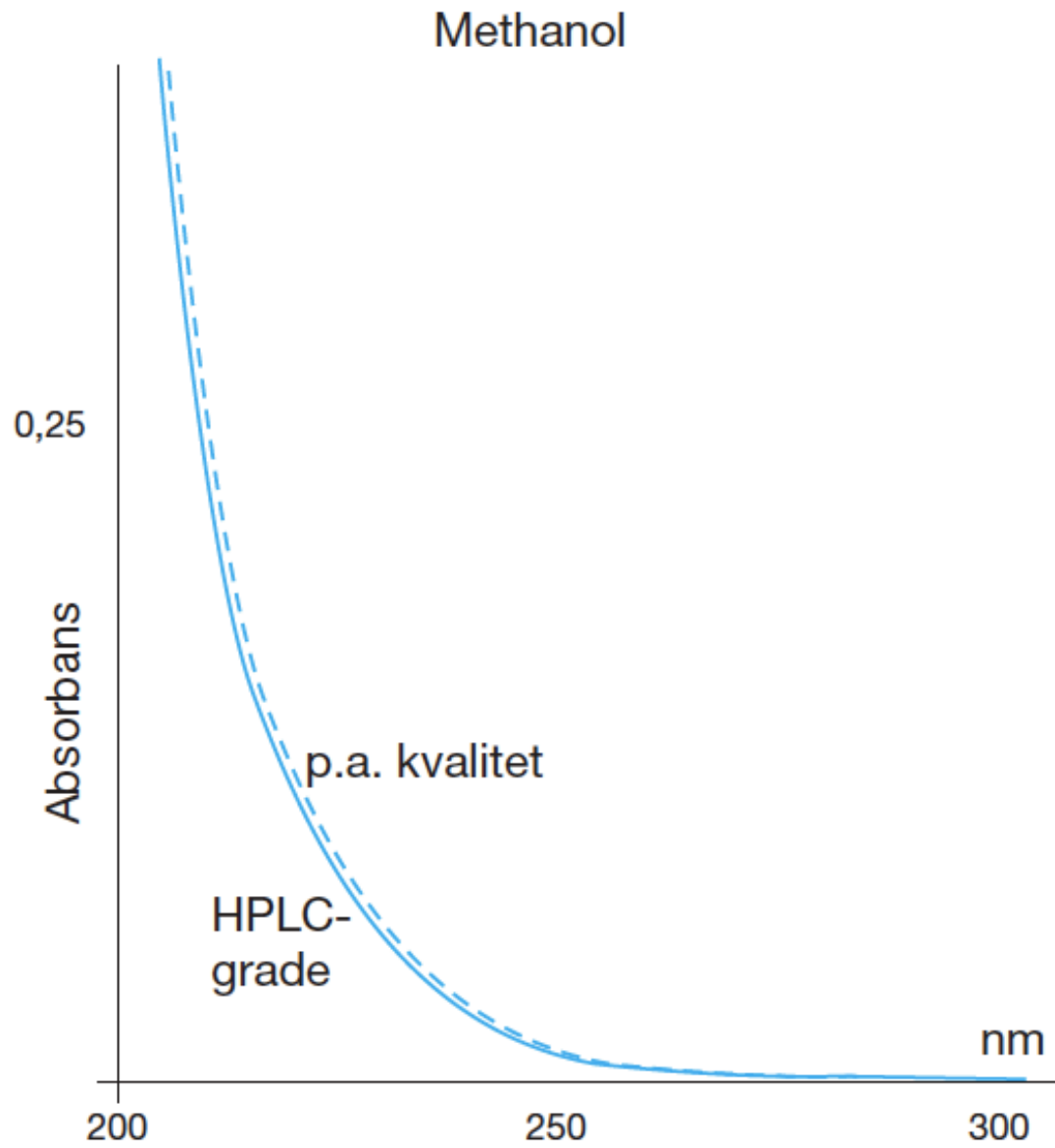
Figur 9.15. Modifieret silikatoverflade.

<i>Gruppe</i>	<i>Navn</i>	<i>Betegnelsen</i>
$\text{Si}(\text{CH}_2)_{17}\text{CH}_3$	Octadecyl-	ODS/C18
$\text{Si}(\text{CH}_2)_3\text{-NH}_2$	Amin-	NH_2
$\text{Si}(\text{CH}_2)_3\text{-CN}$	Cyano-/ nitril-	CN
$\text{Si-C}_6\text{F}_5$	Pentafluorphenyl	PFP
Si-OH	Silica-	Si

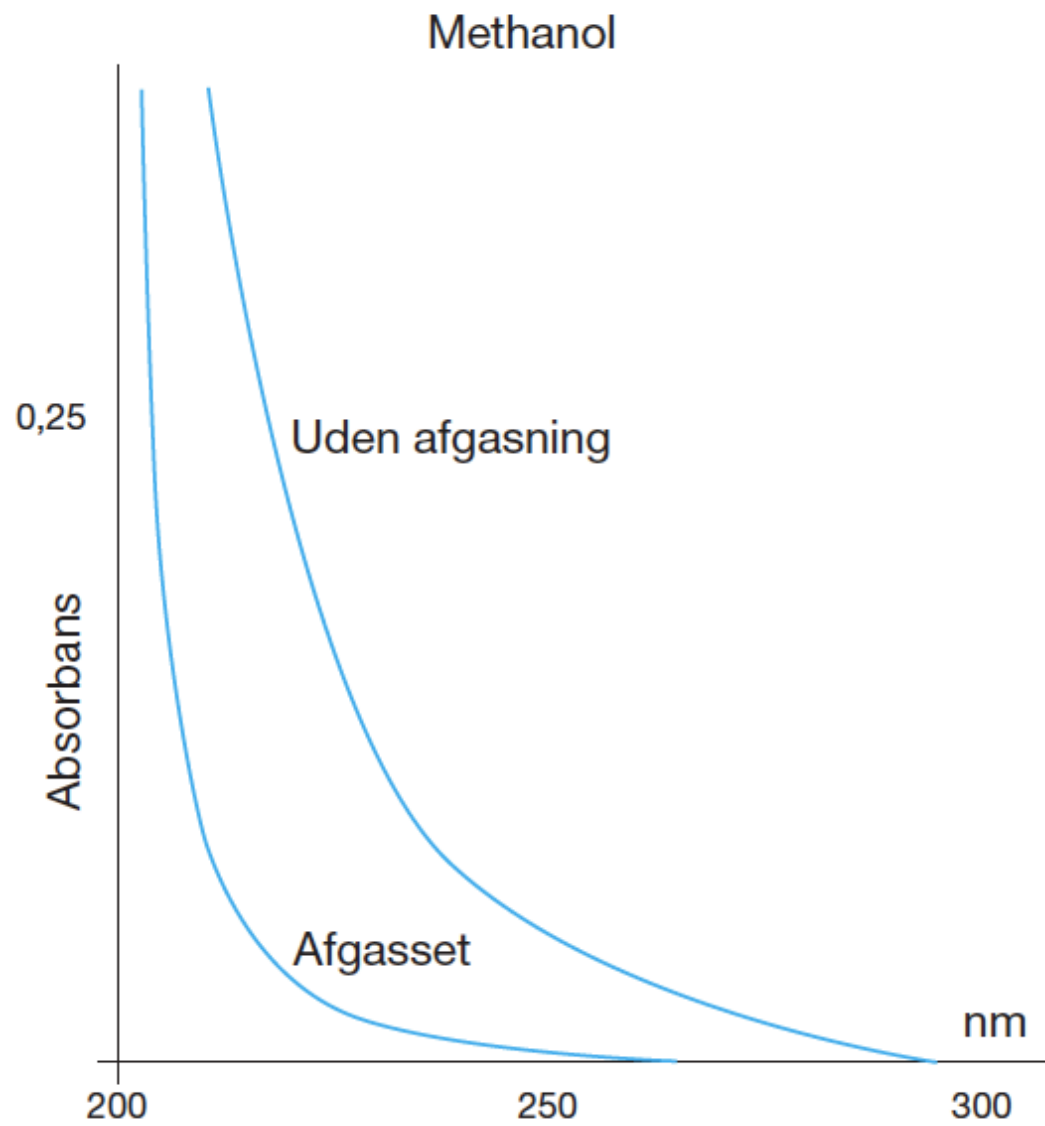
Tabel 9.2.



Figur 9.16. UV-cut off for acetonitril.



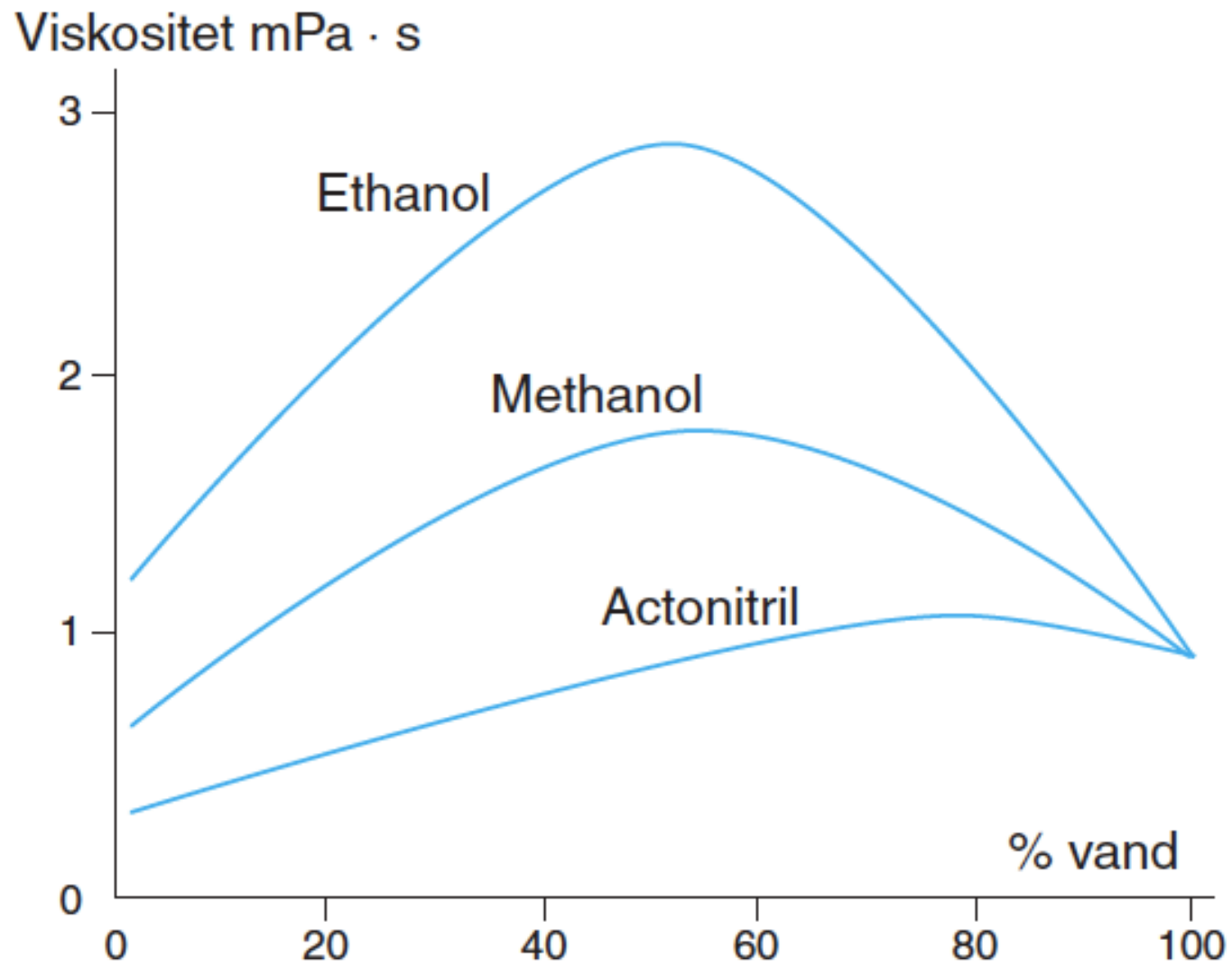
Figur 9.17. UV-cut off for methanol.



Figur 9.18. Afgasningens indflydelse på UV-cut off.

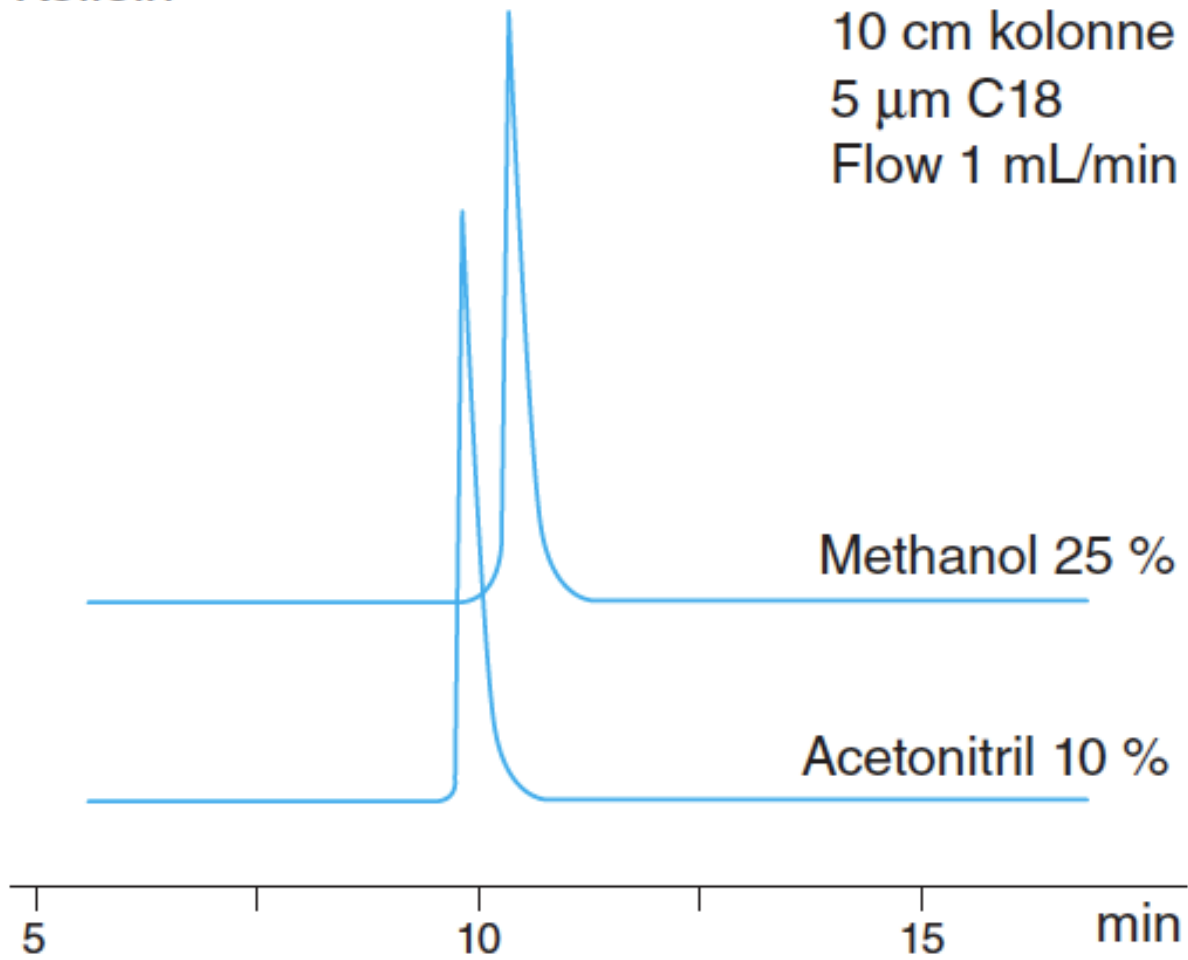
<i>Eluent</i>	<i>Eluerings- evne⁸⁵ 1/ε⁰</i>	<i>Viskositet (mPa · s)</i>	<i>UV-cutoff</i>
n-hexan	100	0,30	200
Toluen	3,4	0,59	285
Tetrahydrofuran	2,2	0,46	215
Acetone	1,8	0,32	330
Acetonitril	1,5	0,37	190
Propan-2-ol	1,2	2,30	210
Ethanol	1,1	1,20	210
Methanol	1,0	0,60	195
Vand	lille	1,00	

Tabel 9.3.



Figur 9.19. Viskositet ved varierende vandindhold.

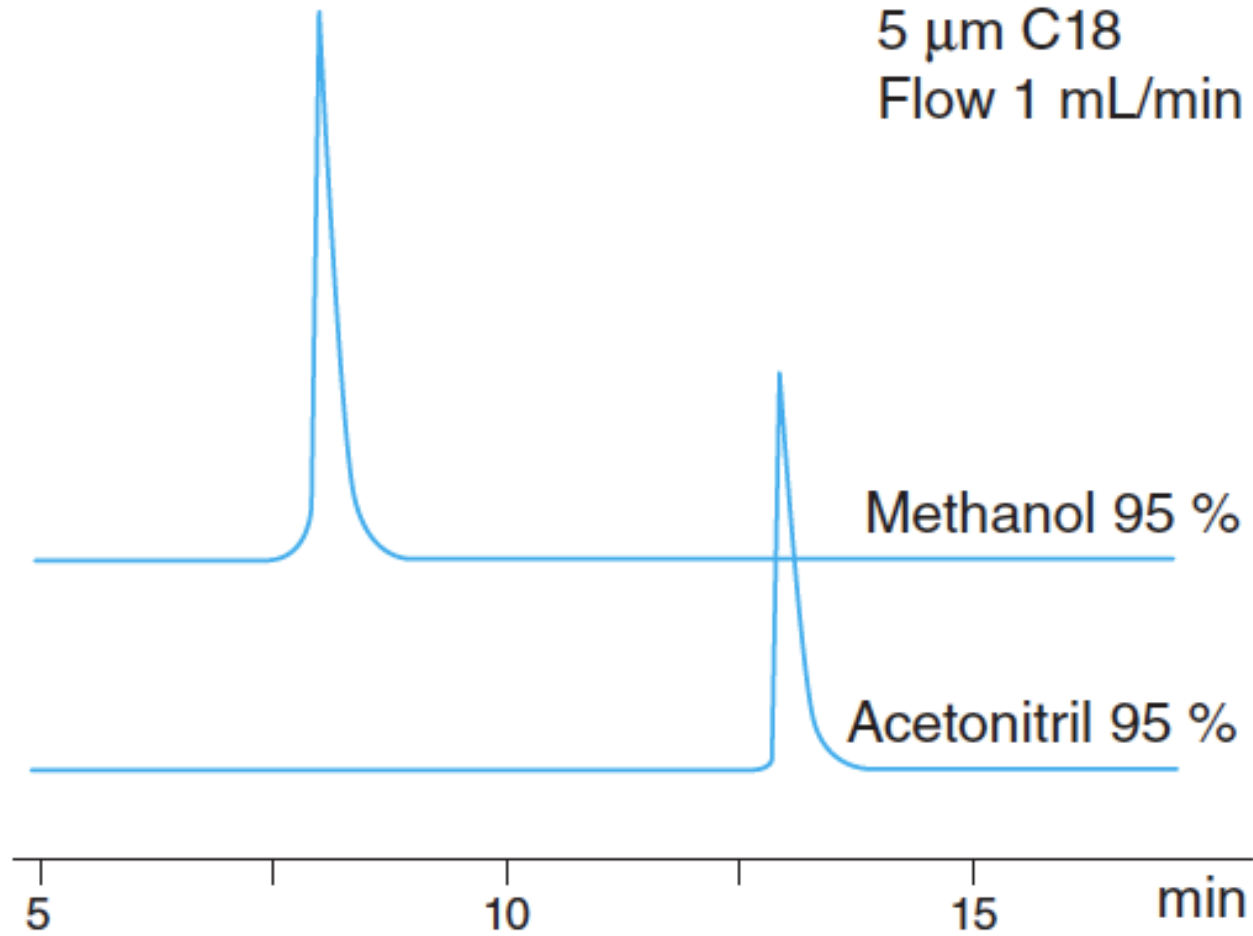
Koffein



Figur 9.20. Sammenligning af elueringssevne for acetonitril og methanol.

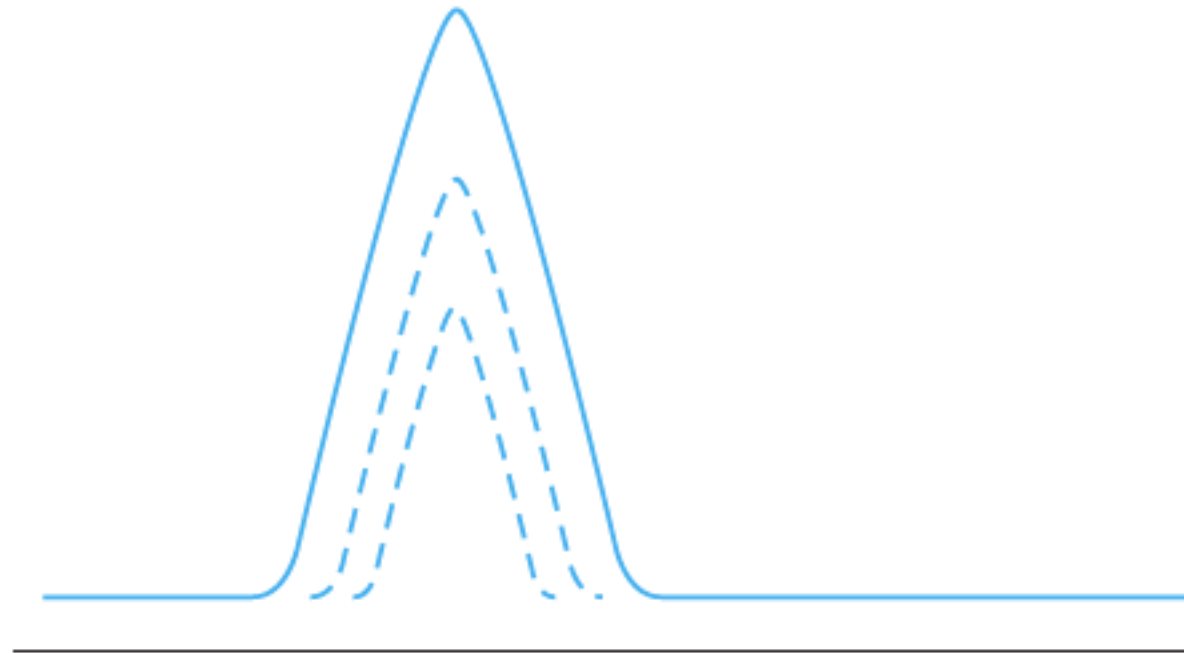
Cholesterol

10 cm kolonne
5 μm C18
Flow 1 mL/min



Figur 9.21. Atypisk elueringsevne.

100 % acetonitril



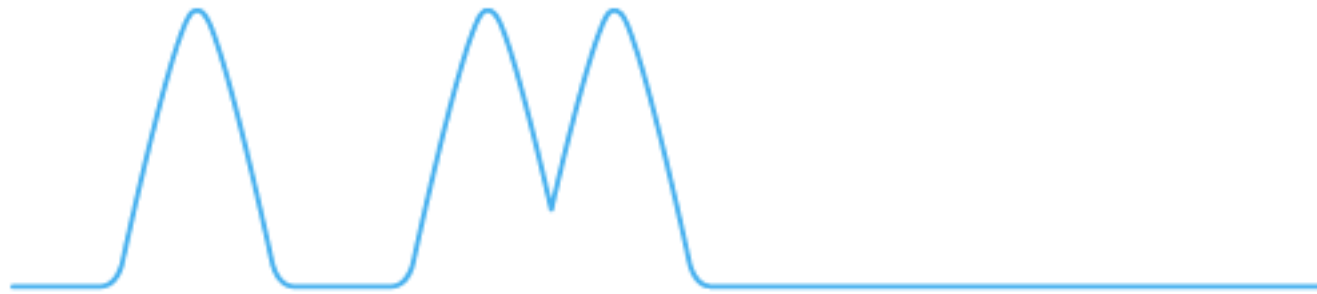
Figur 9.22. Eluering med 100 % acetonitril.

100 % methanol



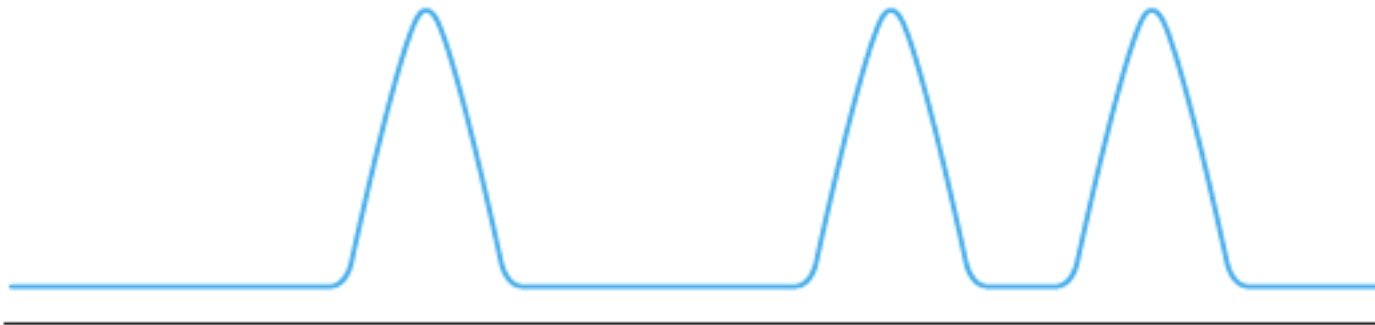
Figur 9.23. Eluering med 100 % methanol.

80 % methanol: 20 % vand



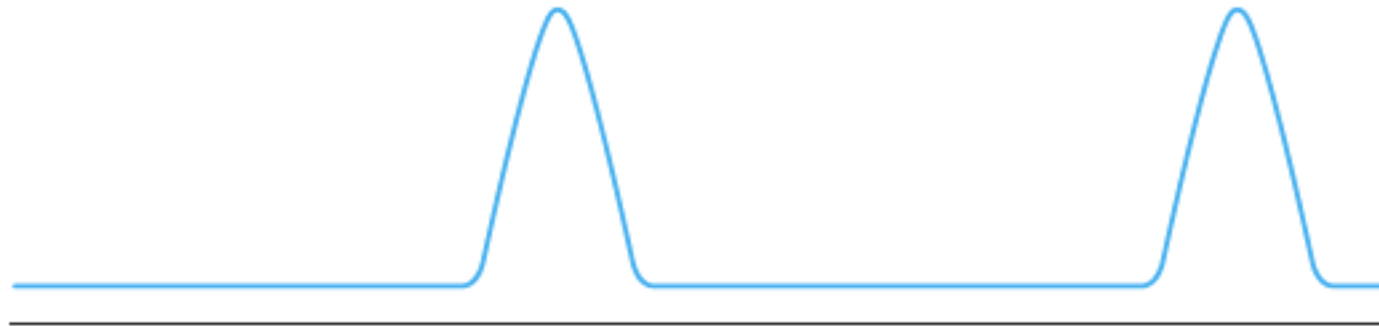
Figur 9.24. Eluering med 80 % methanol.

60 % methanol: 40 % vand

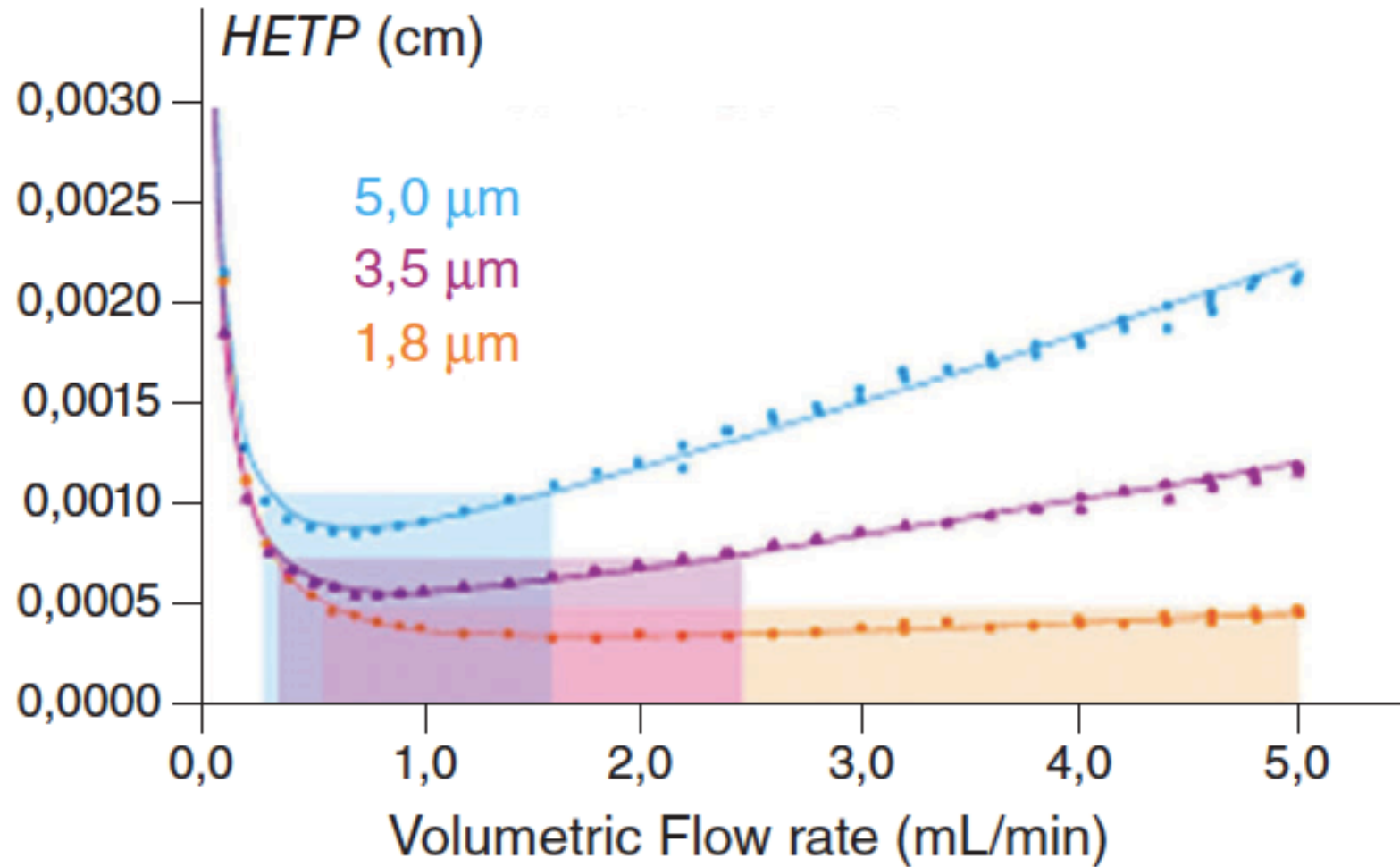


Figur 9.25. Eluering med 60 % methanol

40 % methanol: 60 % vand

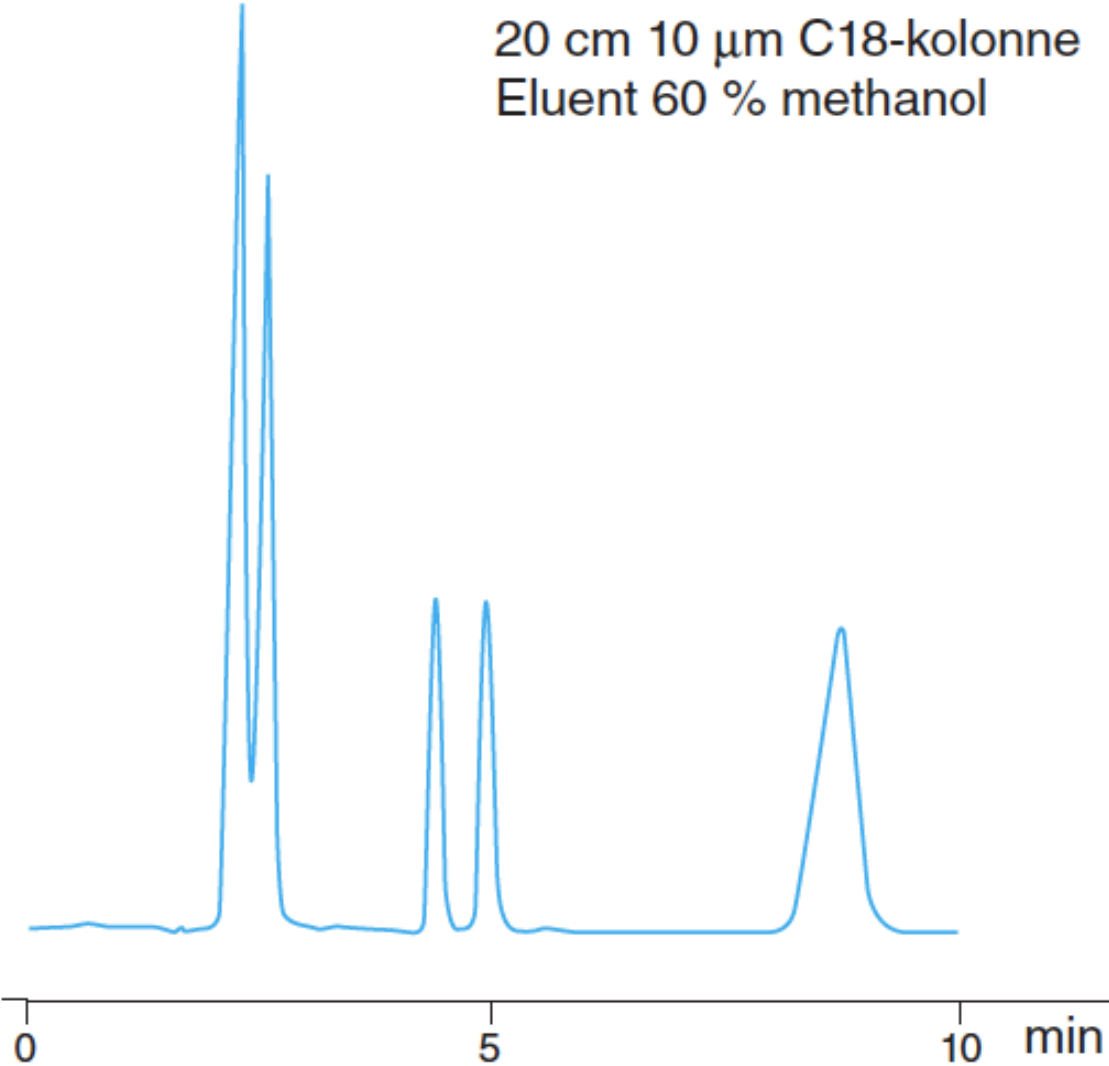


Figur 9.26. Eluering med 40 % methanol.

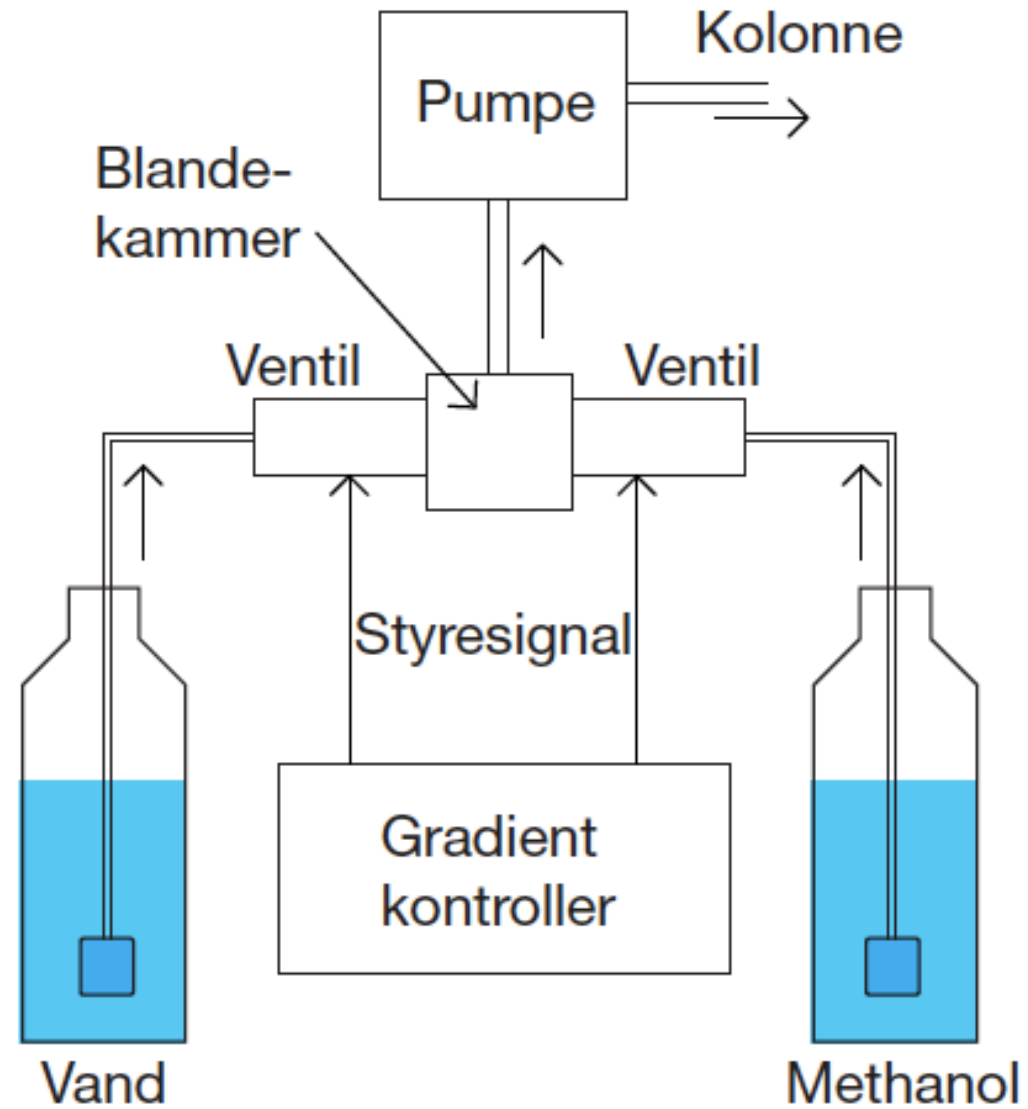


Figur 9.27. Van Deemter-kurve.

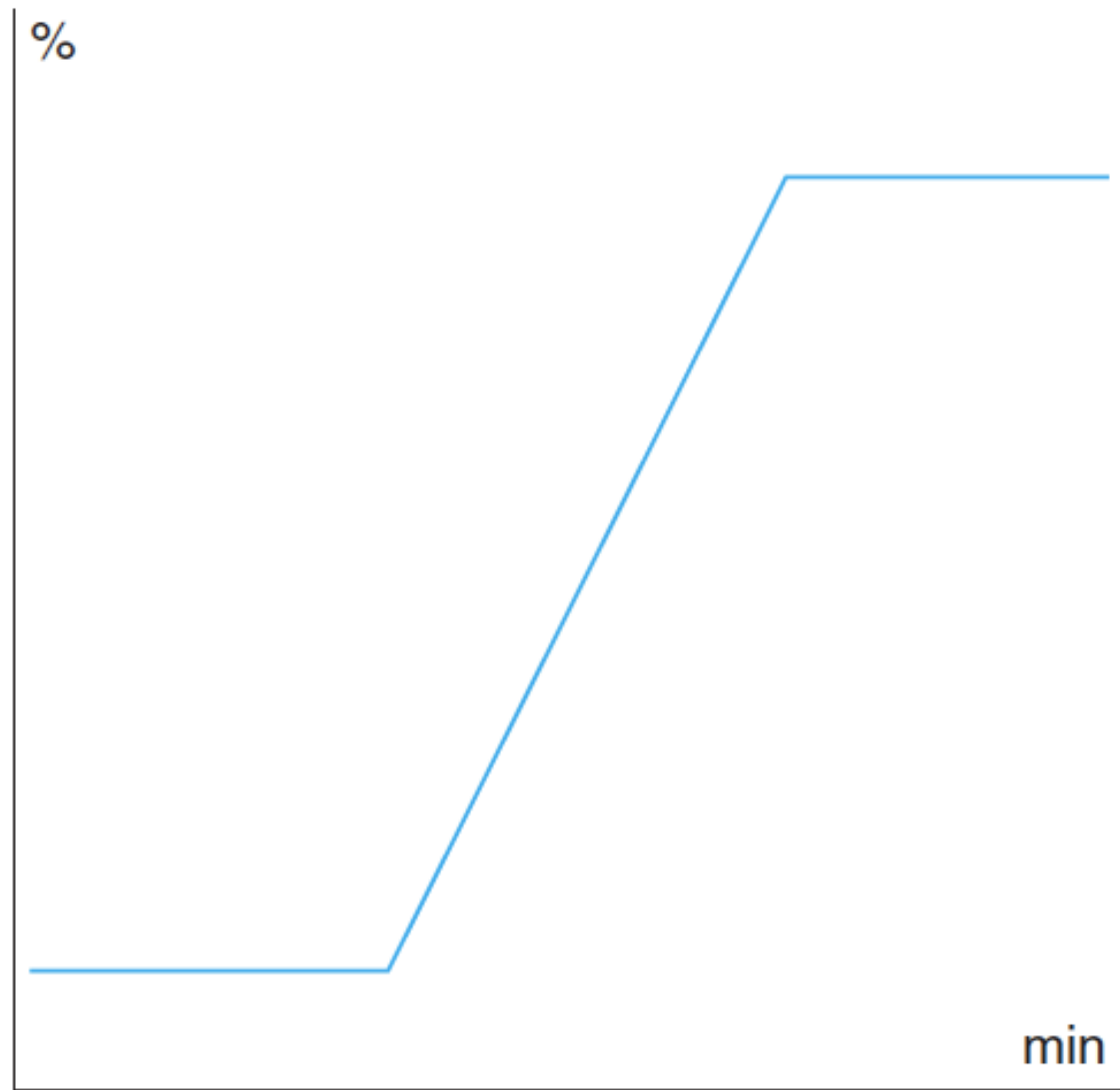
20 cm 10 μ m C18-kolonne
Eluent 60 % methanol



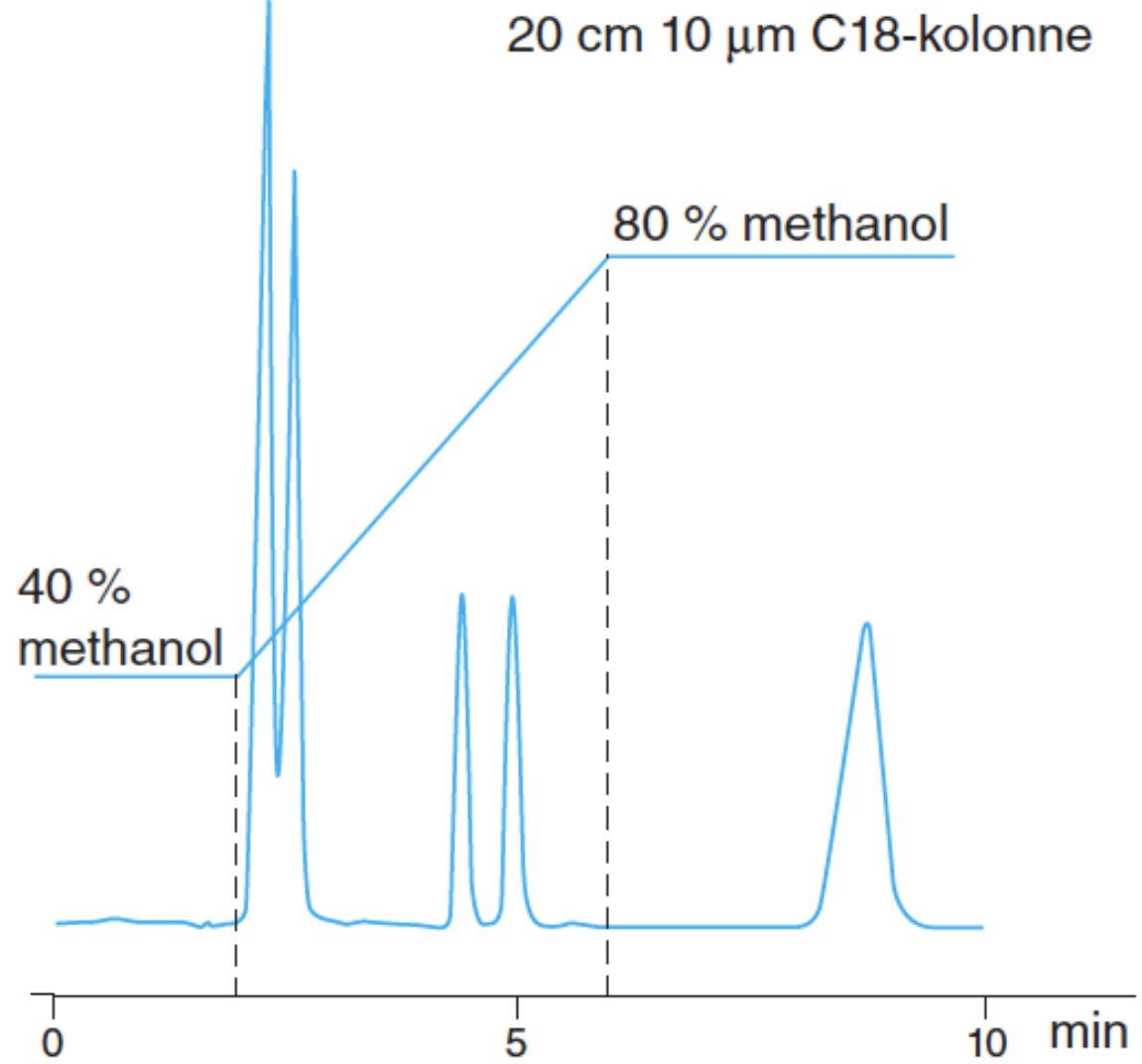
Figur 9.28. Problematisk eluering under isokratiske forhold.



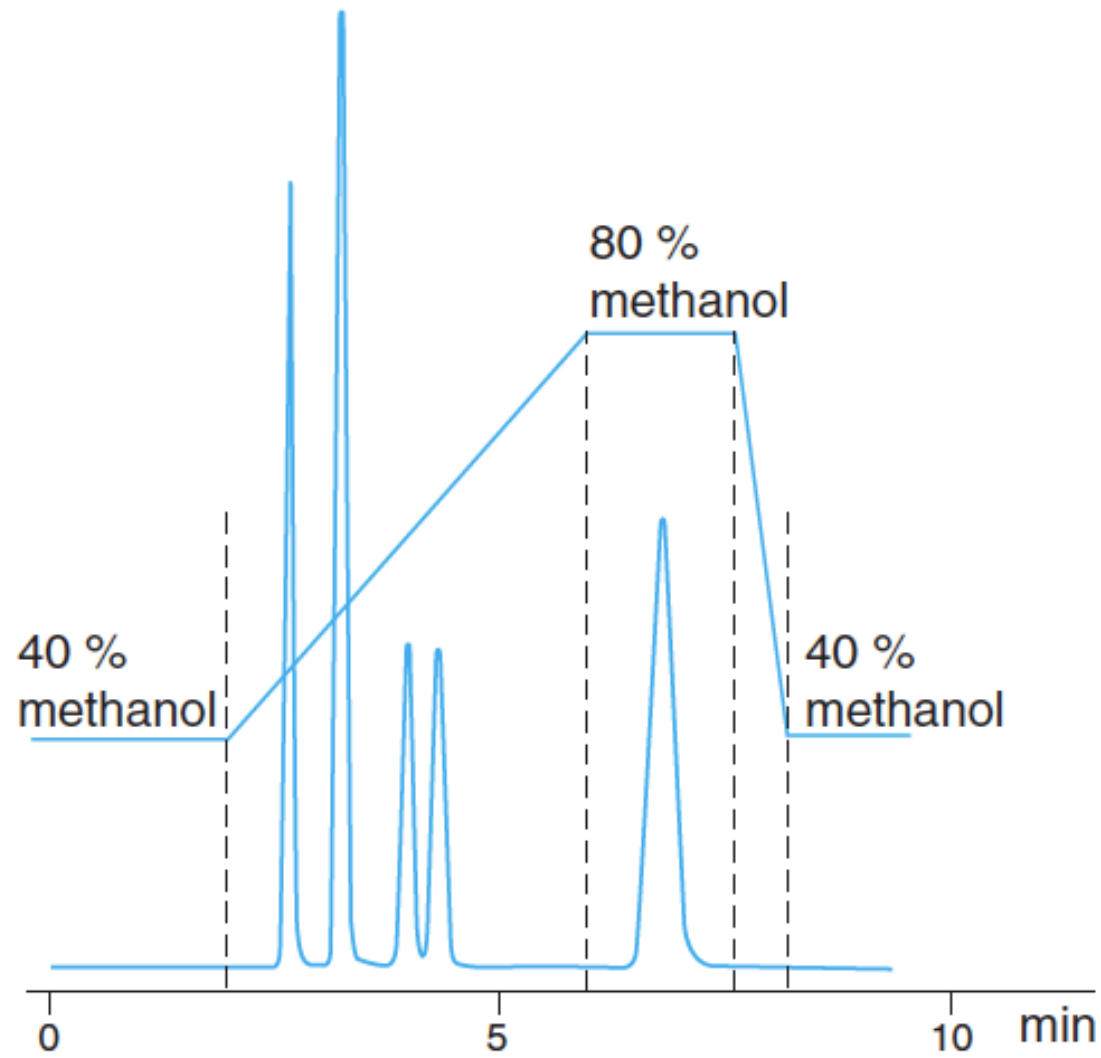
Figur 9.29. Gradientsystemet.



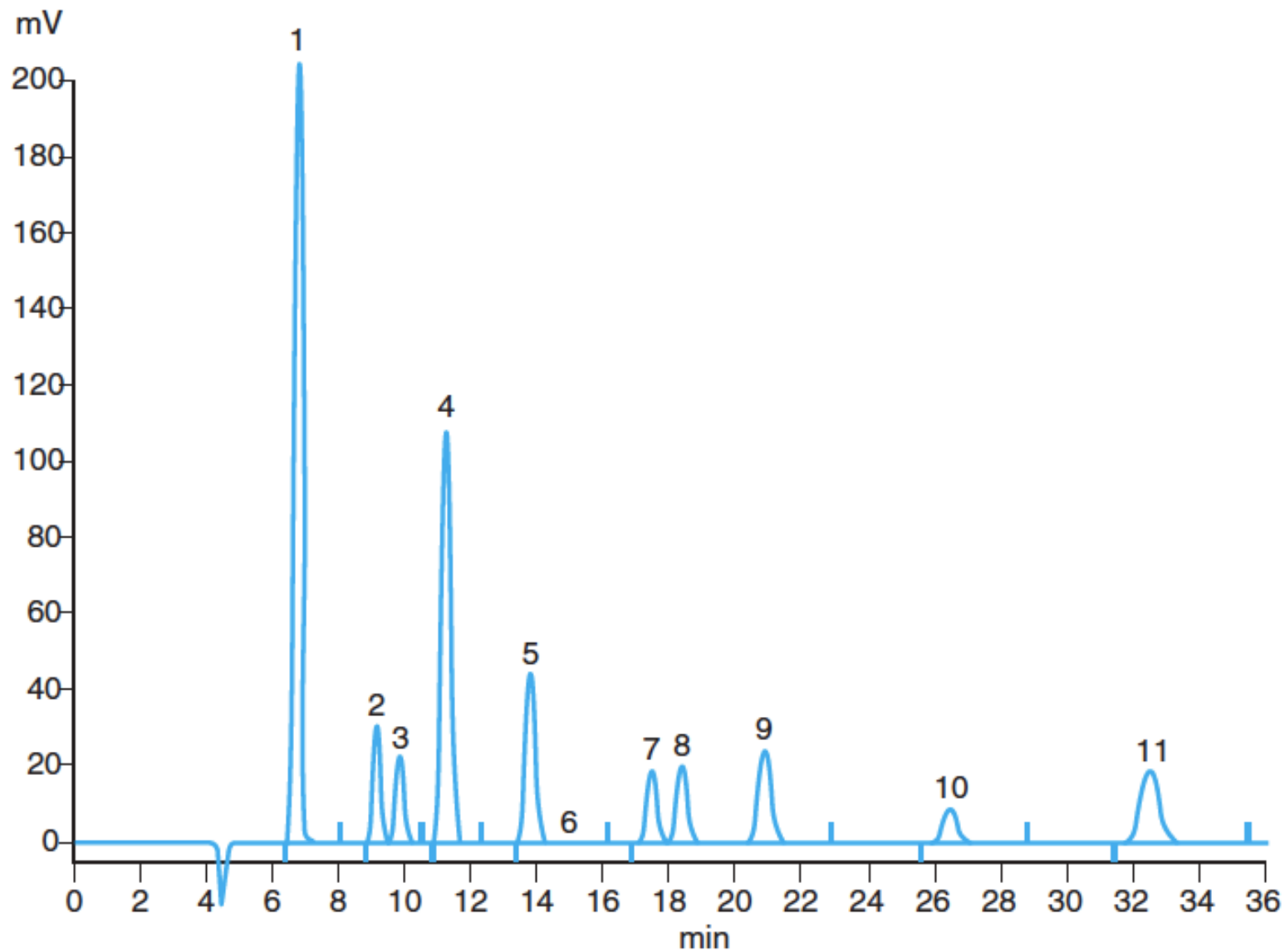
Figur 9.30. Gradientprogram.



Figur 9.31. Forslag til gradientprogram.

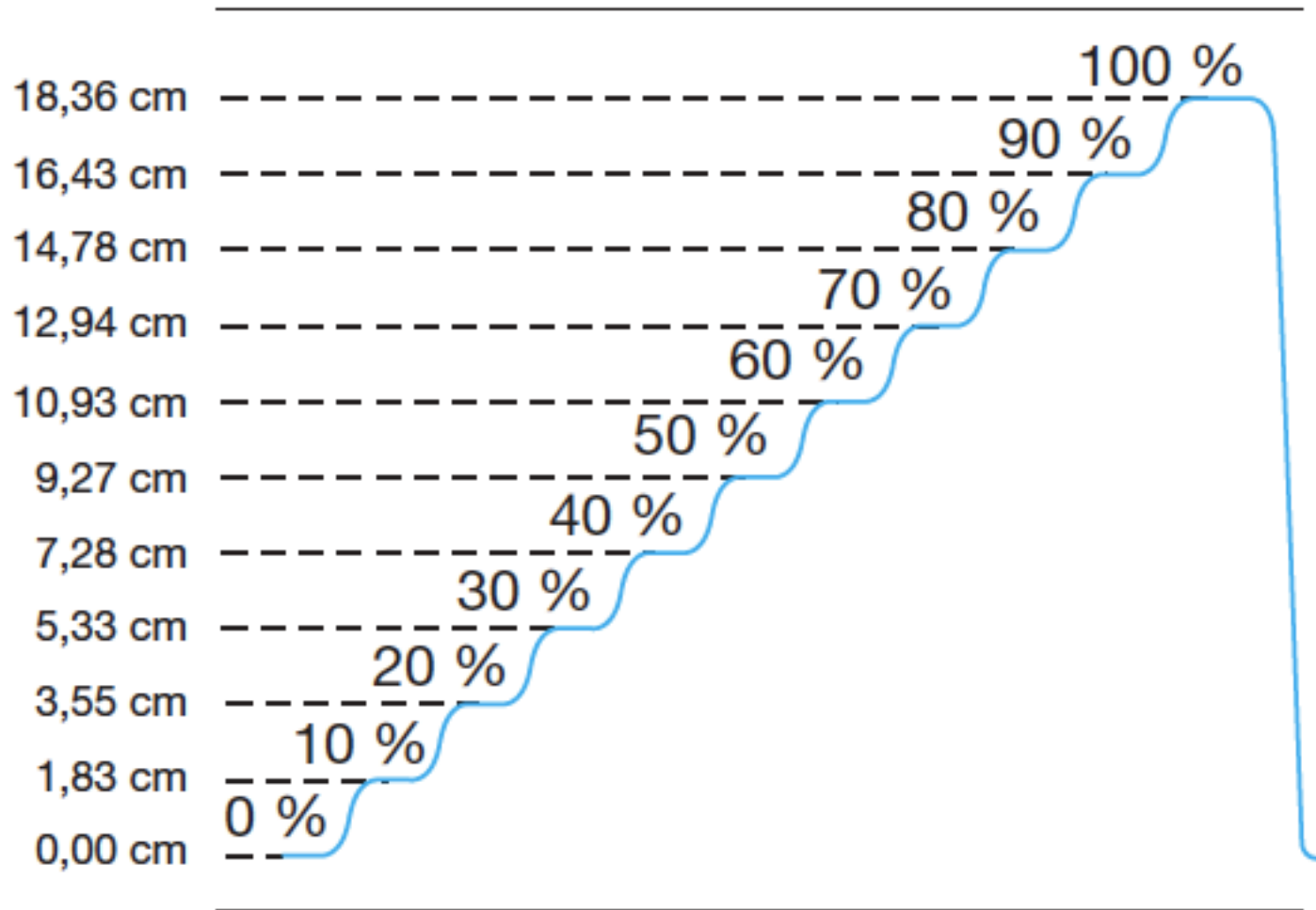


Figur 9.32. Gradienteluering med indtegnet gradient-program.

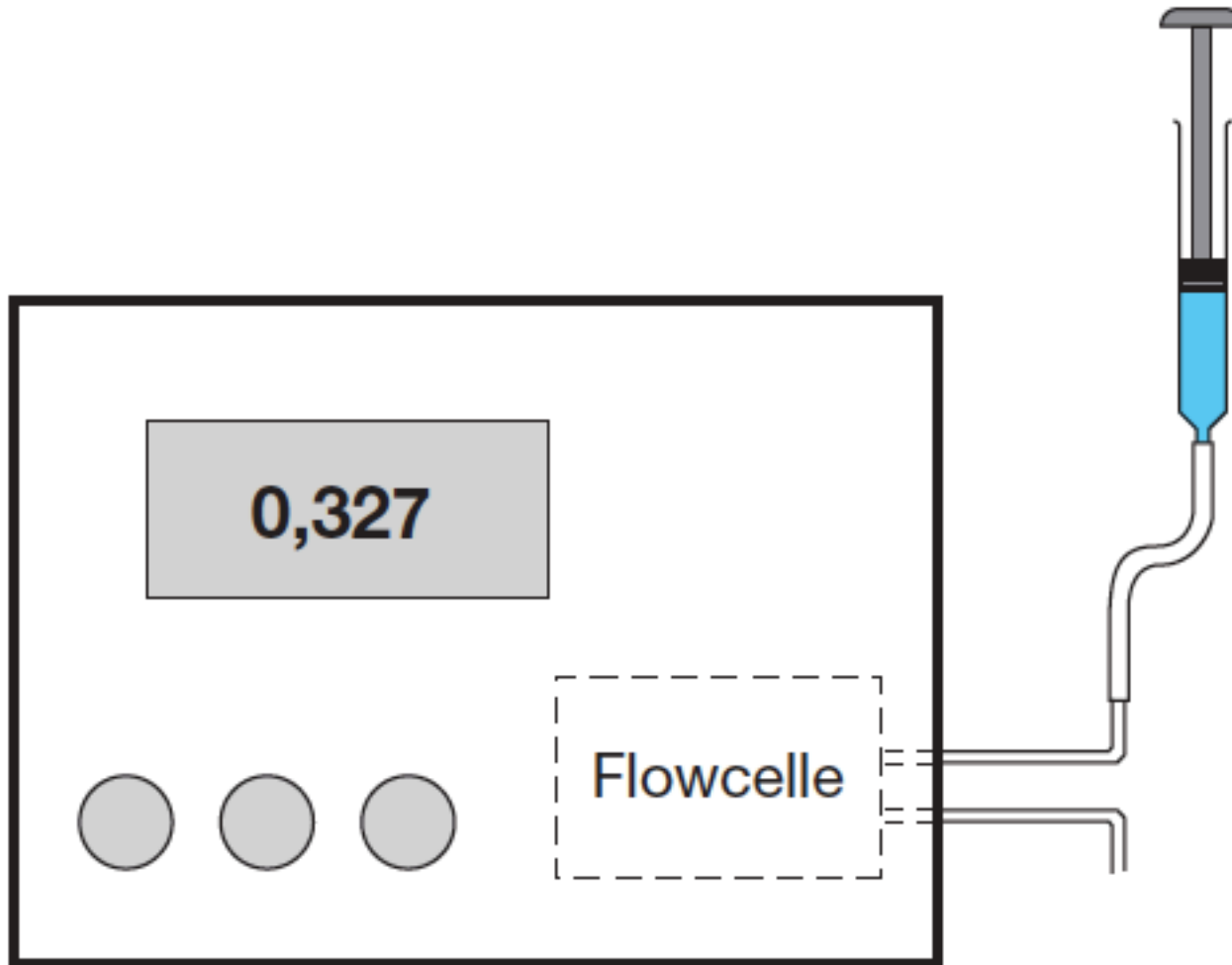


A Supp 5 – 250		
<i>NaHCO₃/Na₂CO₃; 1/3,2</i>		
1	Fluoride	5,0
2	Chlorite	5,0
3	Bromate	5,0
4	Chloride	5,0
5	Nitrite	5,0
7	Chlorate	5,0
8	Bromide	5,0
9	Nitrate	5,0
10	Phosphate	5,0
11	Sulfate	5,0

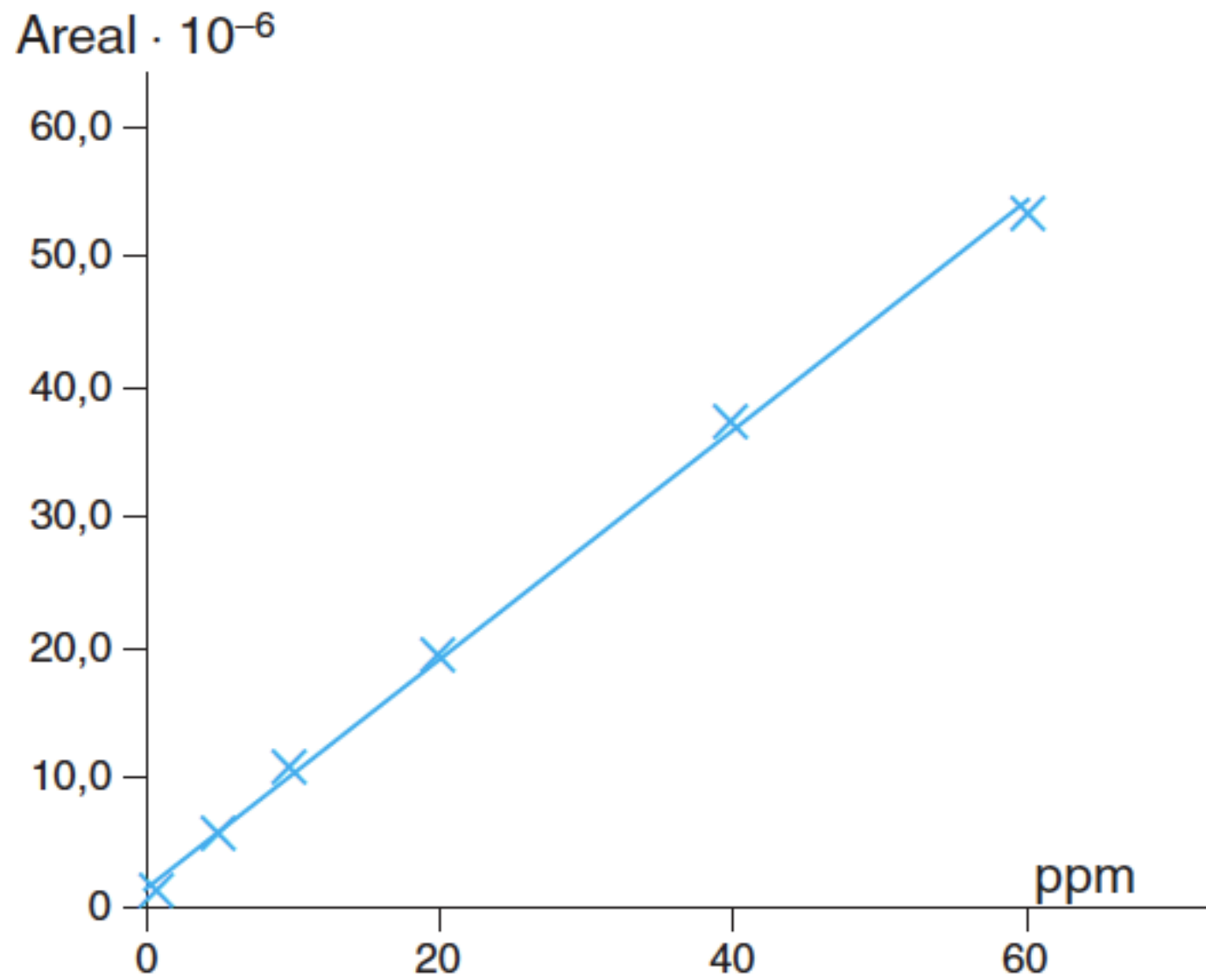
Figur 9.33. Et eksempel på et chromatogram fra en LC-IC bestemmelse af anioner (fra Metrohm).



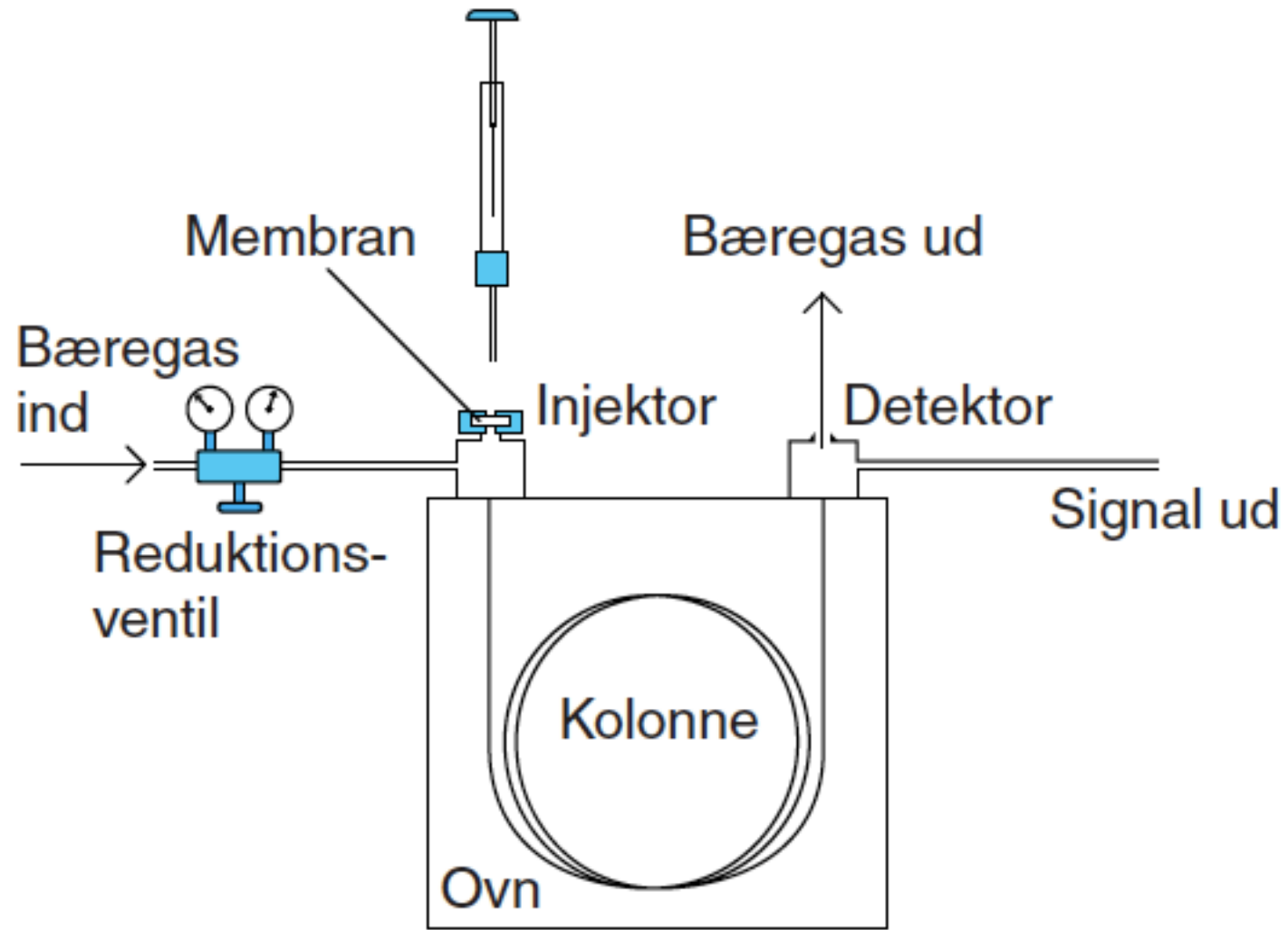
Figur 9.34. Kontrol af gradientblandesystemet.



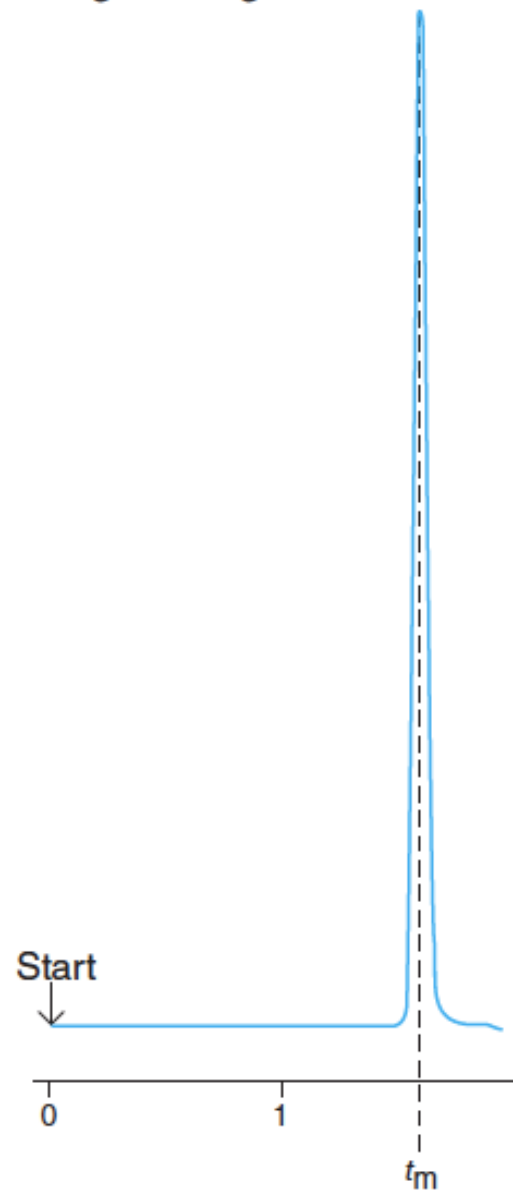
Figur 9.35. Kontrol af UV-detektor.



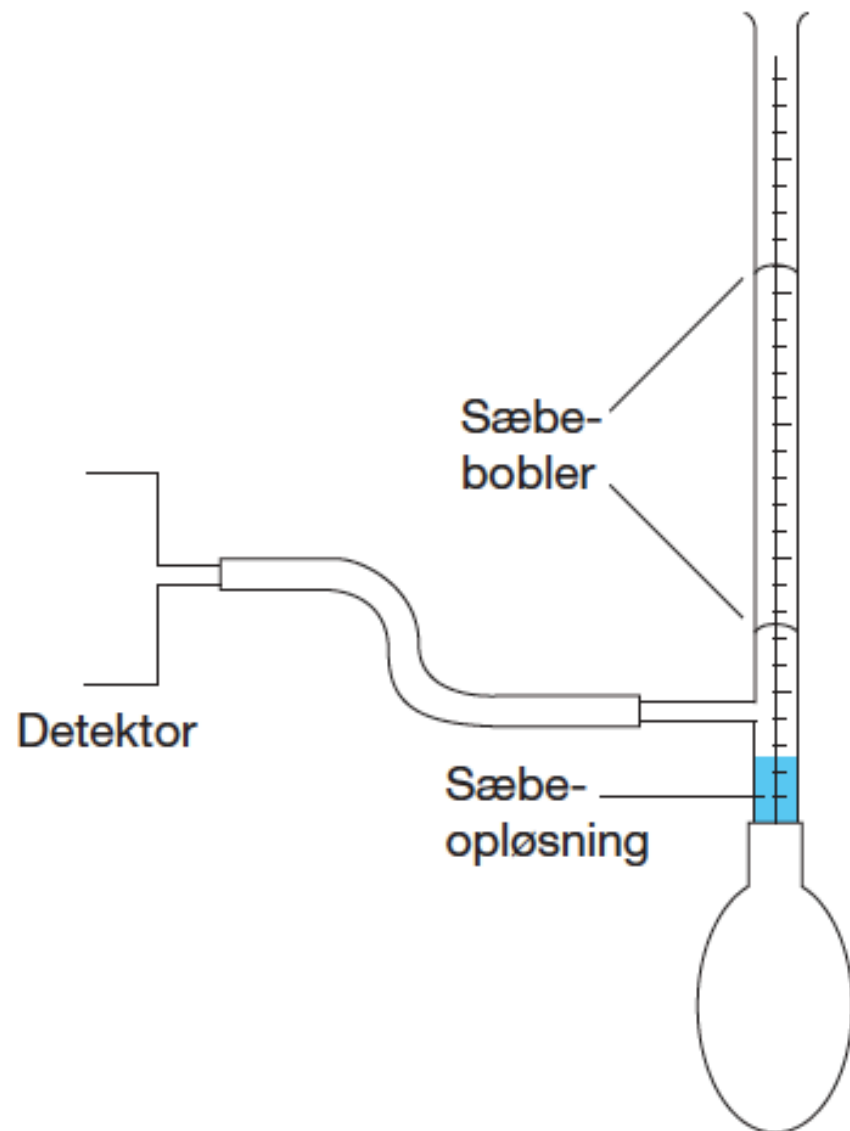
Figur 9.36. Linearitetskontrol, HPLC.



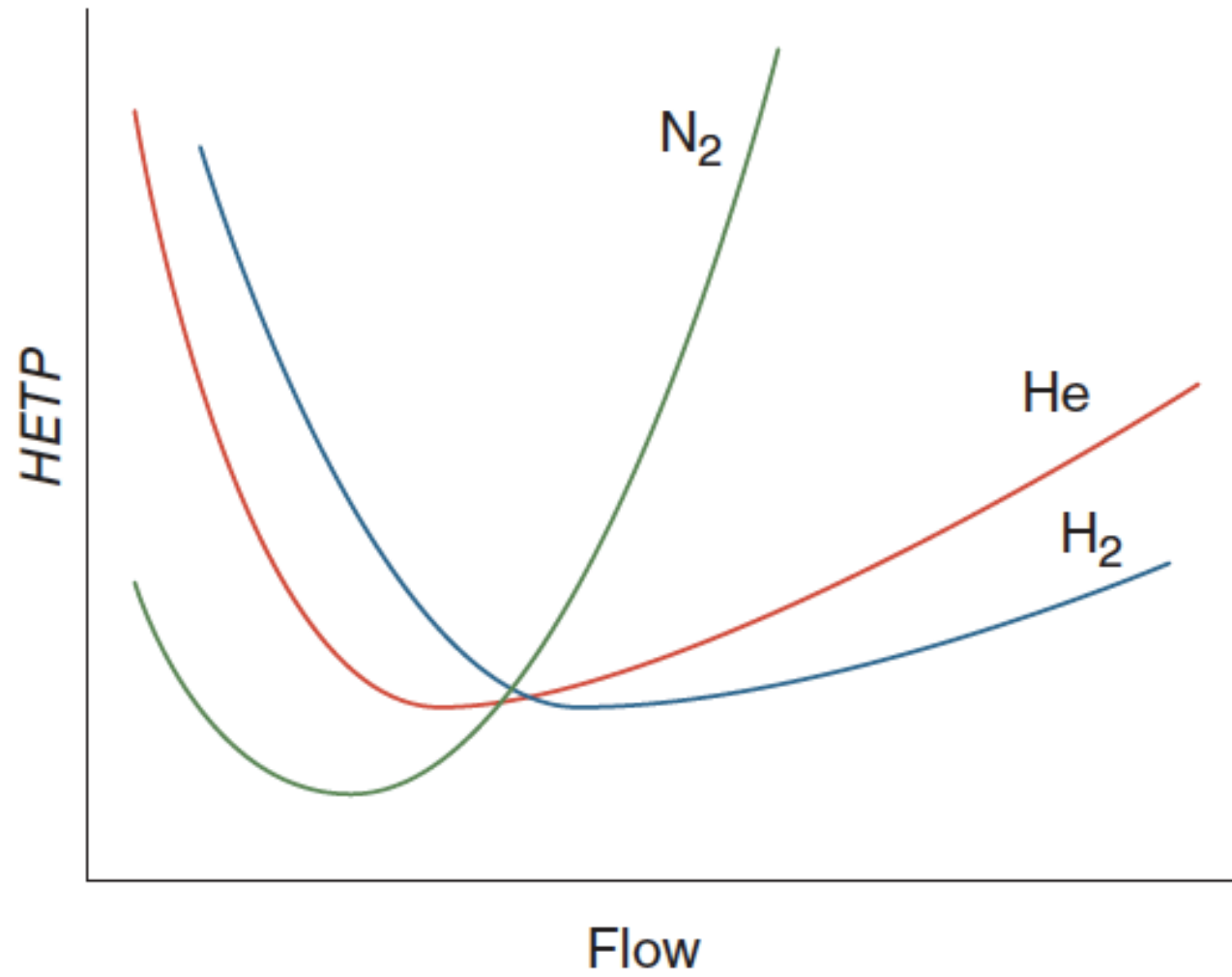
Figur 10.1. GC-systemets opbygning.



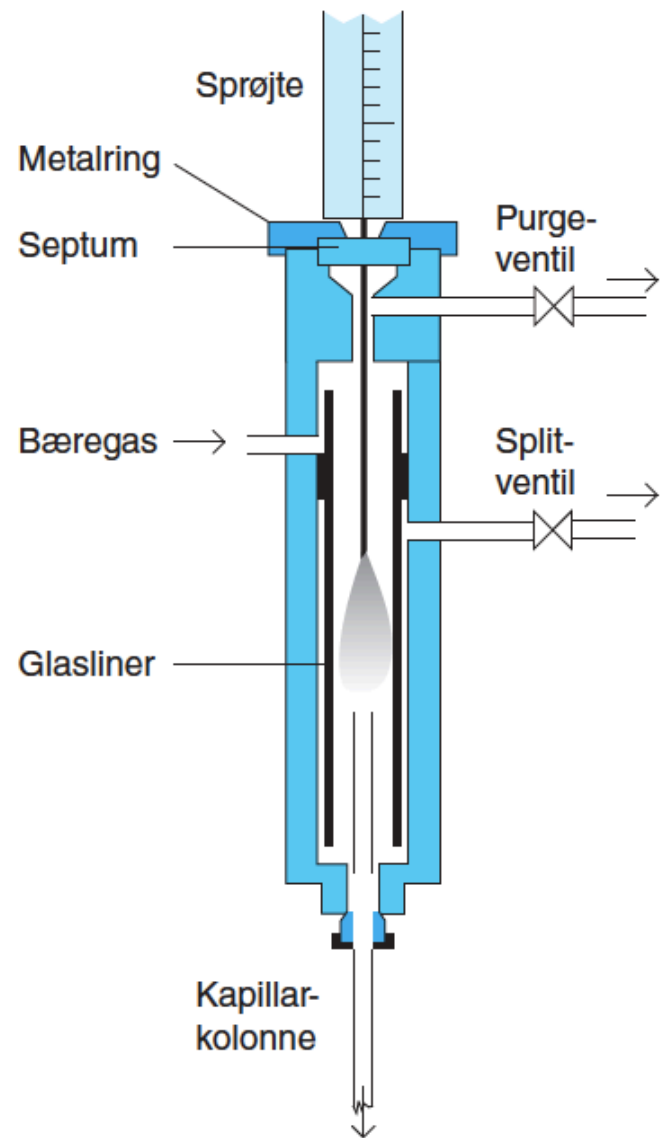
Figur 10.2. Bestemmelse af dødtiden.



Figur 10.3. Sæbebobleflowmeter.



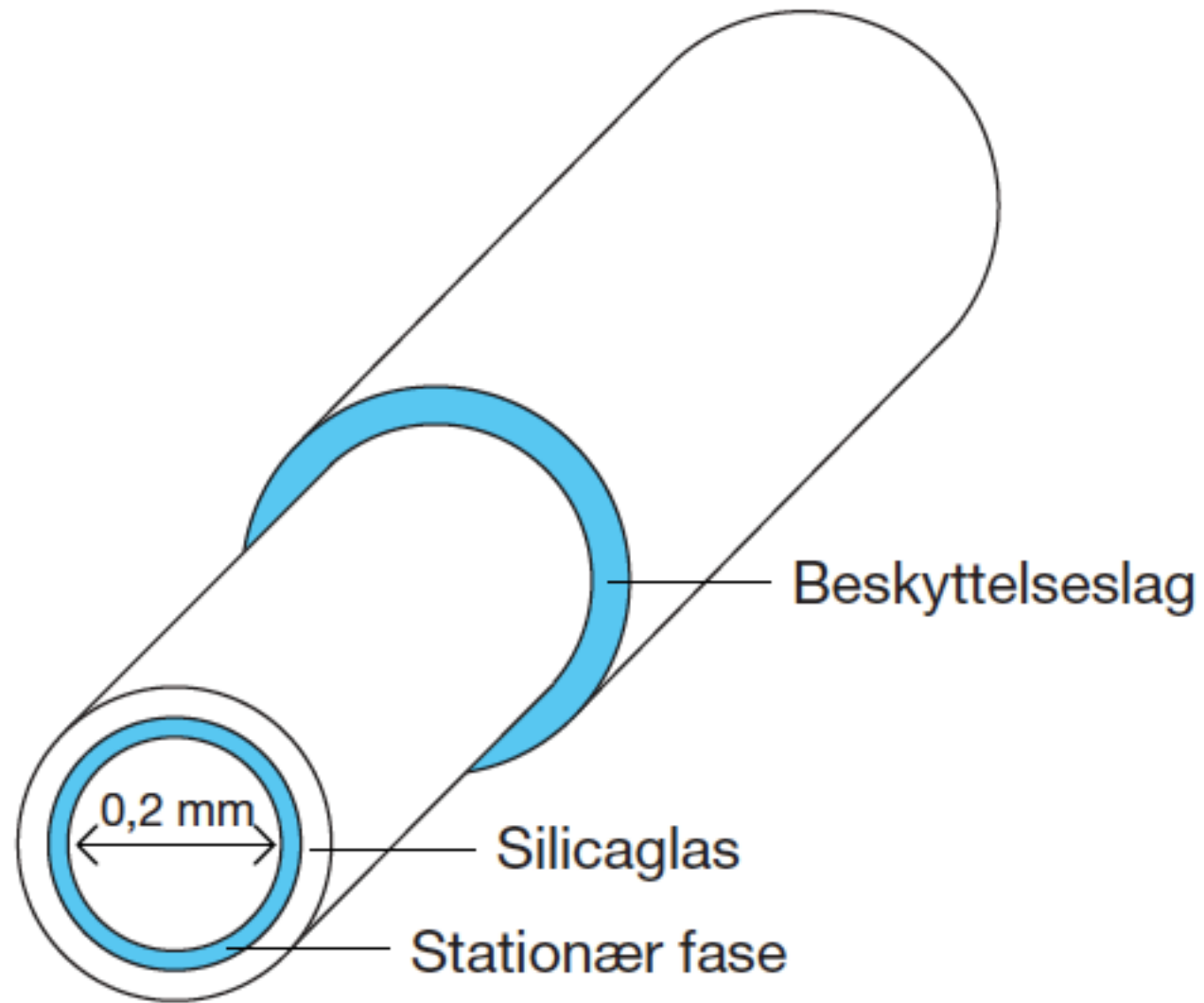
Figur 10.4. Van Deemter-kuve.



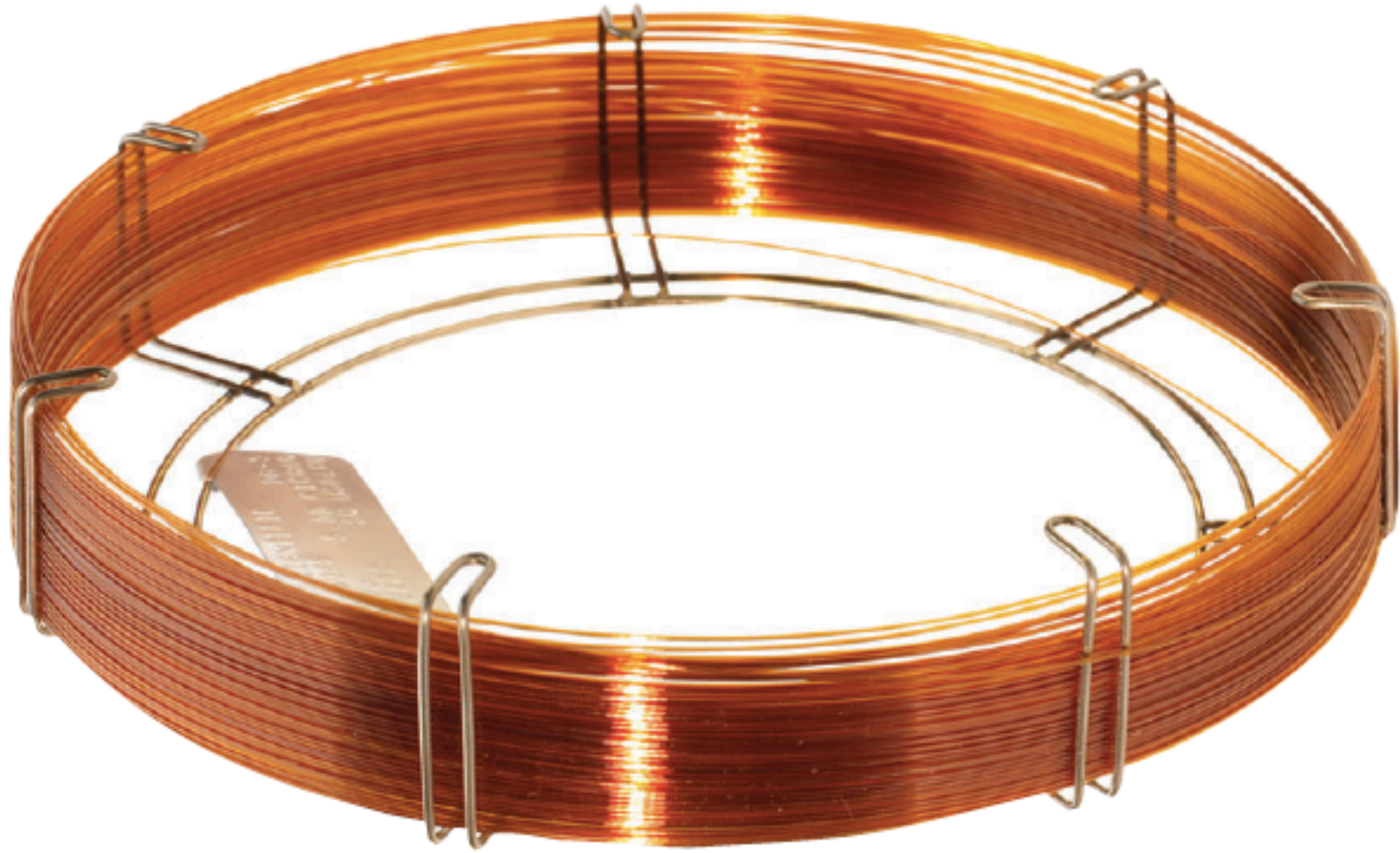
Figur 10.5. Injektor til GC.

<i>Kolonnedata</i>	<i>Kapillar</i>
Længde (m)	5-100
ID (mm)	0,1-0,7
Flow (mL/min)	0,5-15
Bundantal	250000
Kapacitet ($\mu\text{g}/\text{top}$)	0,1
Filmtykkelse (μm)	0,1-8

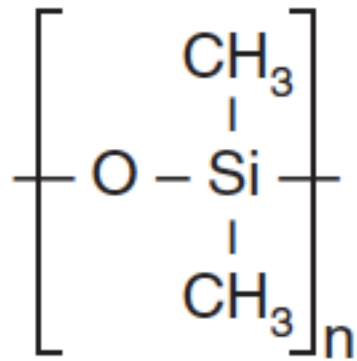
Tabel 10.1. Typiske kolonnedata.



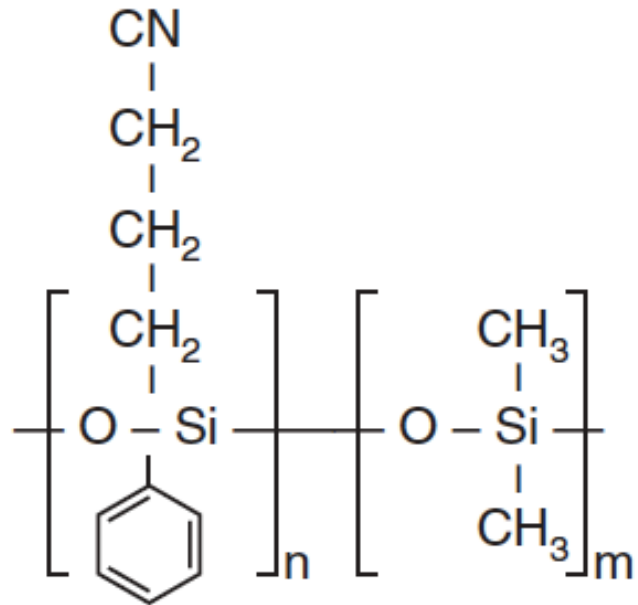
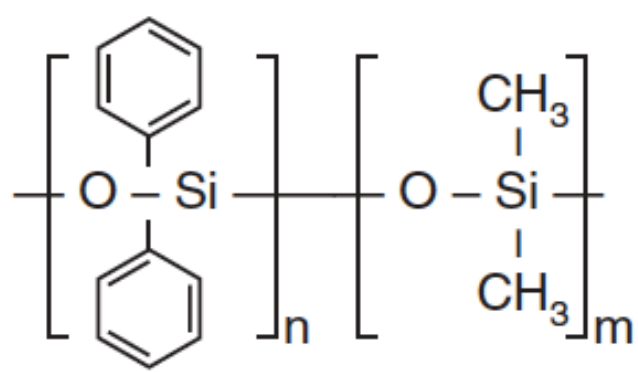
Figur 10.6. Nærbillede af kapillarkolonne.



Figur 10.7. Kapillarkolonne viklet op på metalholder.



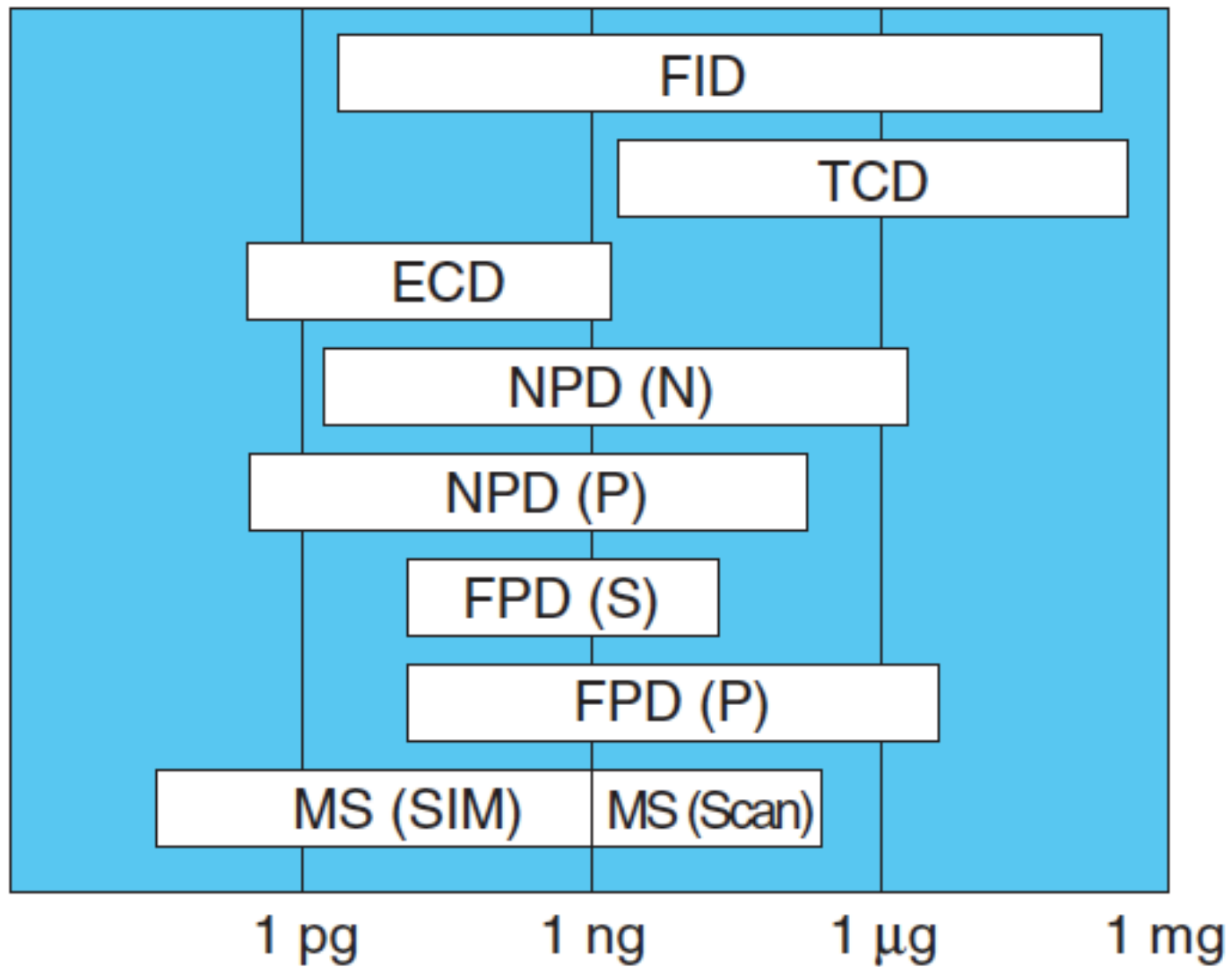
Figur 10.9. Stationær fase i en methyl-kolonne.



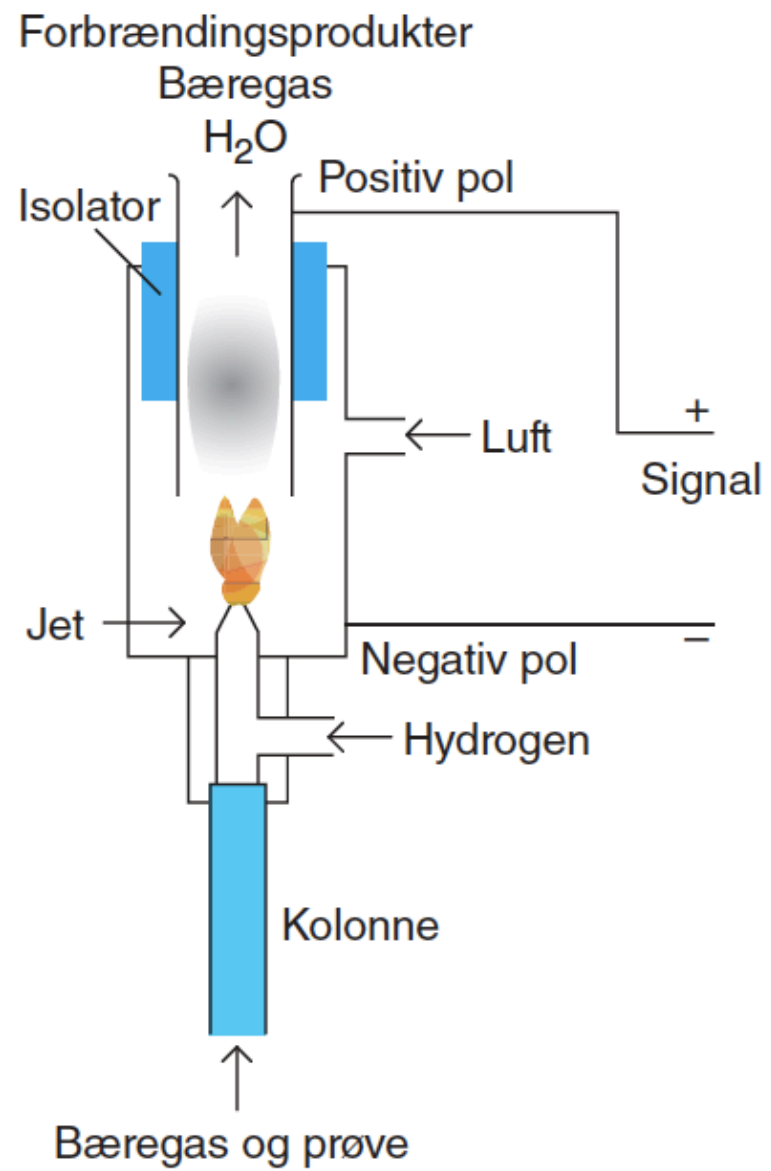
Figur 10.10. Kolonnemateriale bestående af phenyl- og methylgrupper (øverst) eller bestående af cyanopropyl-, phenyl- og methylgrupper

<i>Type</i>	<i>Princip</i>	<i>Forbindelser</i>	<i>LOD</i>	<i>Gasser</i>
FID	Flammeionisering	Organiske	10^{-11} g	H ₂ , luft og enten He eller N ₂
TCD	Termisk konduktivitet	Generel detektor	10^{-9} g	Ofte He
ECD	Elektron Capture	Halogenholdige	10^{-9} g	He og N ₂ eller 5% methan i Ar
NPD	Nitrogen Phosphor	N- og P-holdige	10^{-11} g	H ₂ , luft og enten He eller N ₂
FPD	Flammefotometri	S- og P-holdige	10^{-11} g (S) 10^{-12} g (P)	H ₂ , luft og N ₂
MS	Massespektrometri	Organiske	10^{-12} g	He

Tabel 10.2. Oversigt over forskellige detektortypers egenskaber.

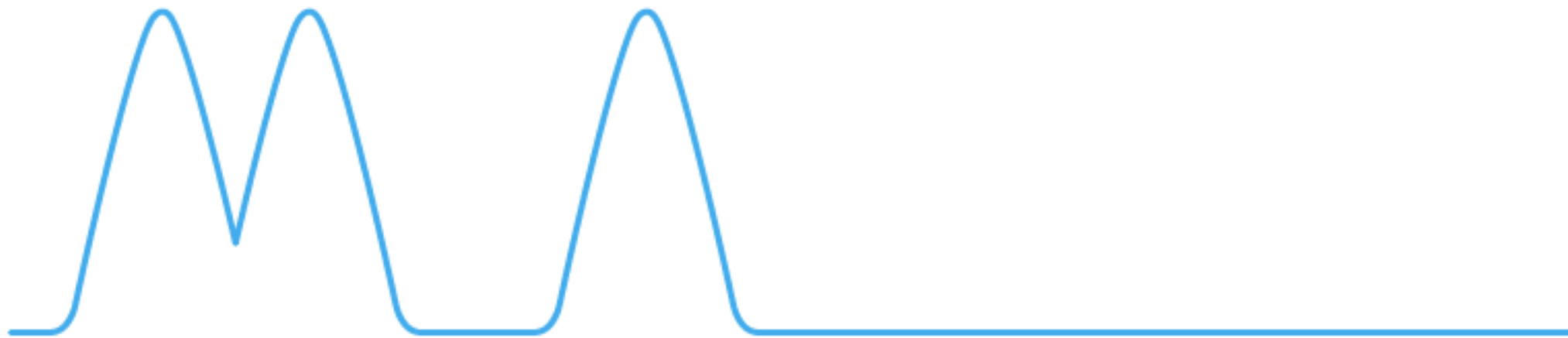


Figur 10.11. Linearitetsområde for udvalgte GC-detektorer.



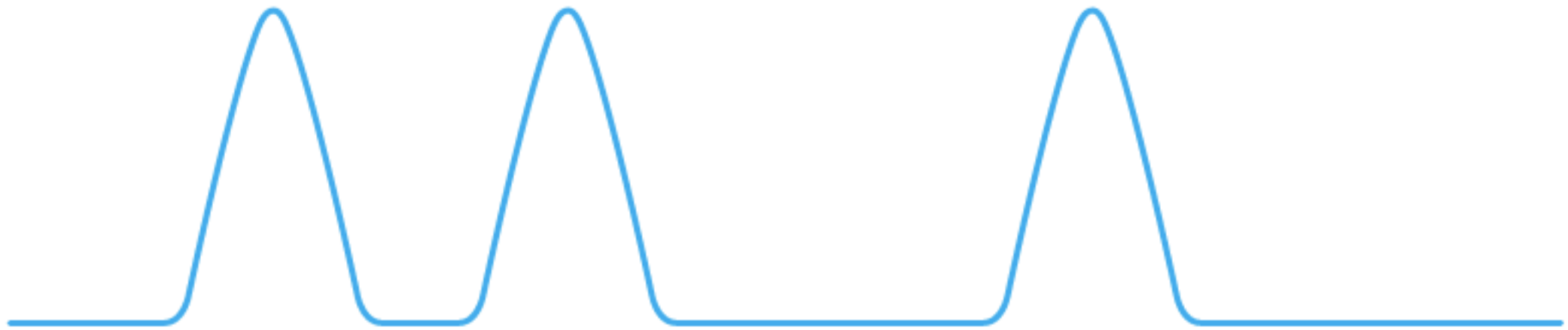
Figur 10.12. Flammeionisationsdetektor (FID).

140 °C



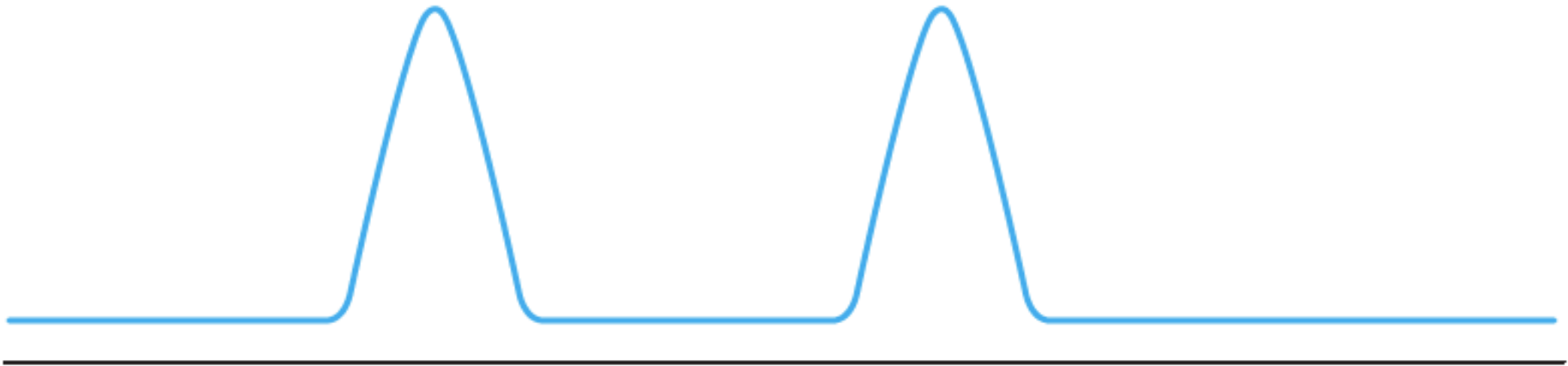
Figur 10.13. Topadskillelse, for høj temperatur.

120 °C

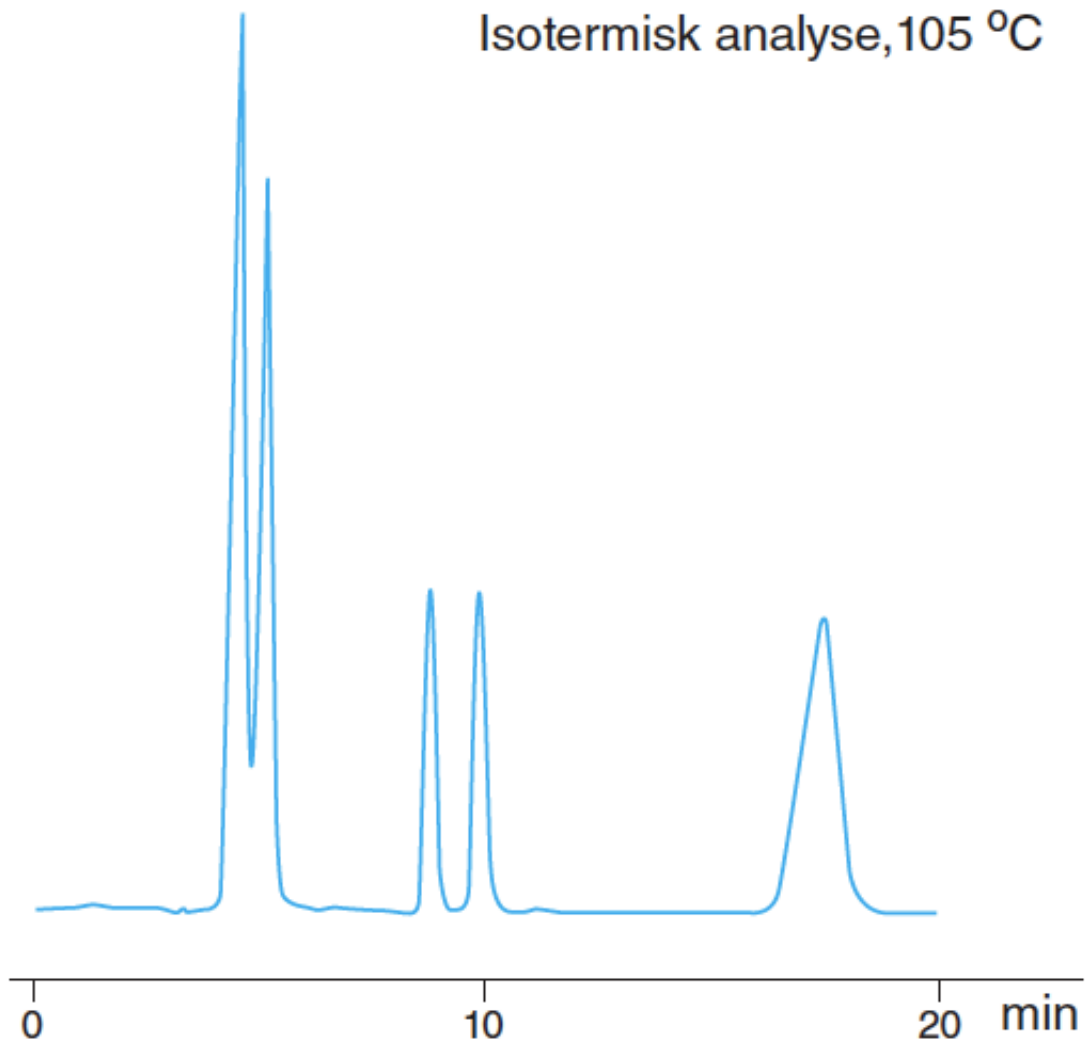


Figur 10.14. Topadskillelse, tilpas temperatur.

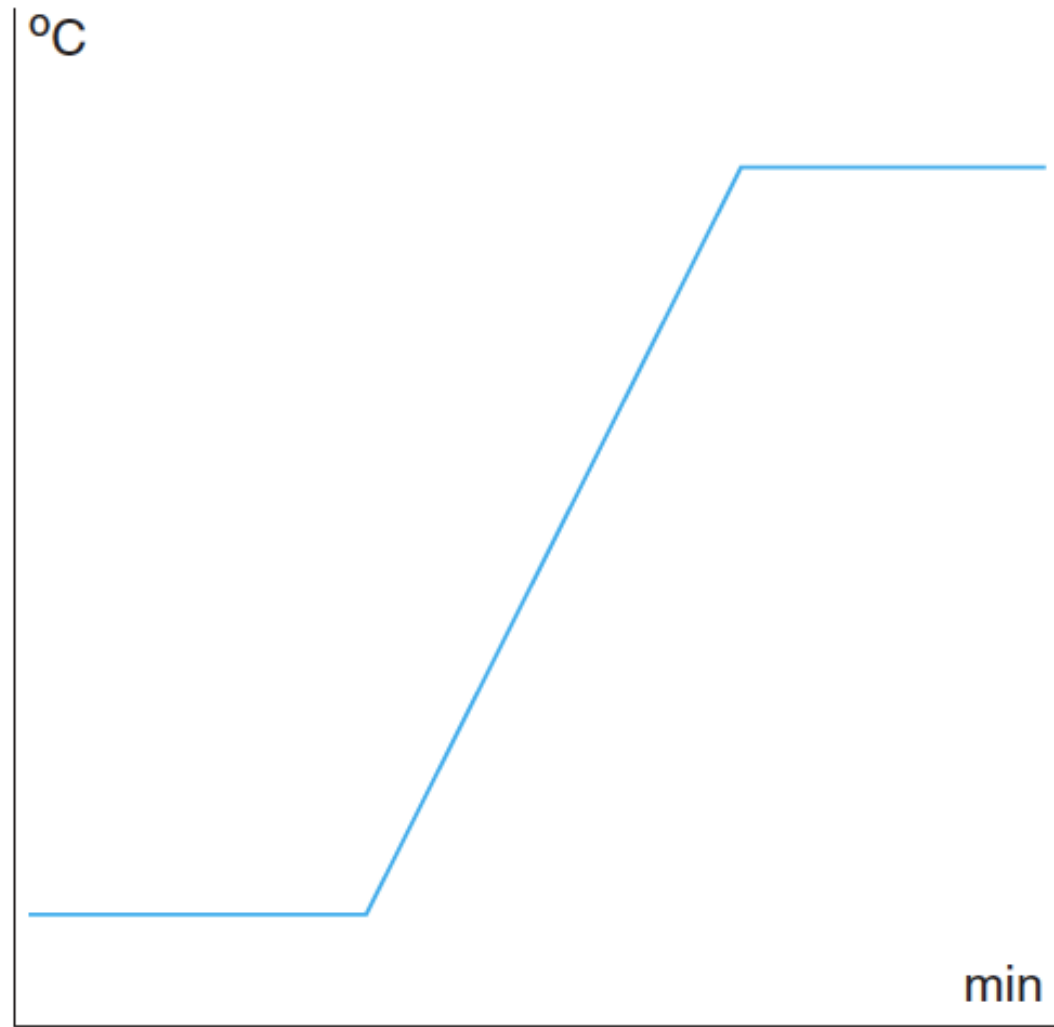
100 °C



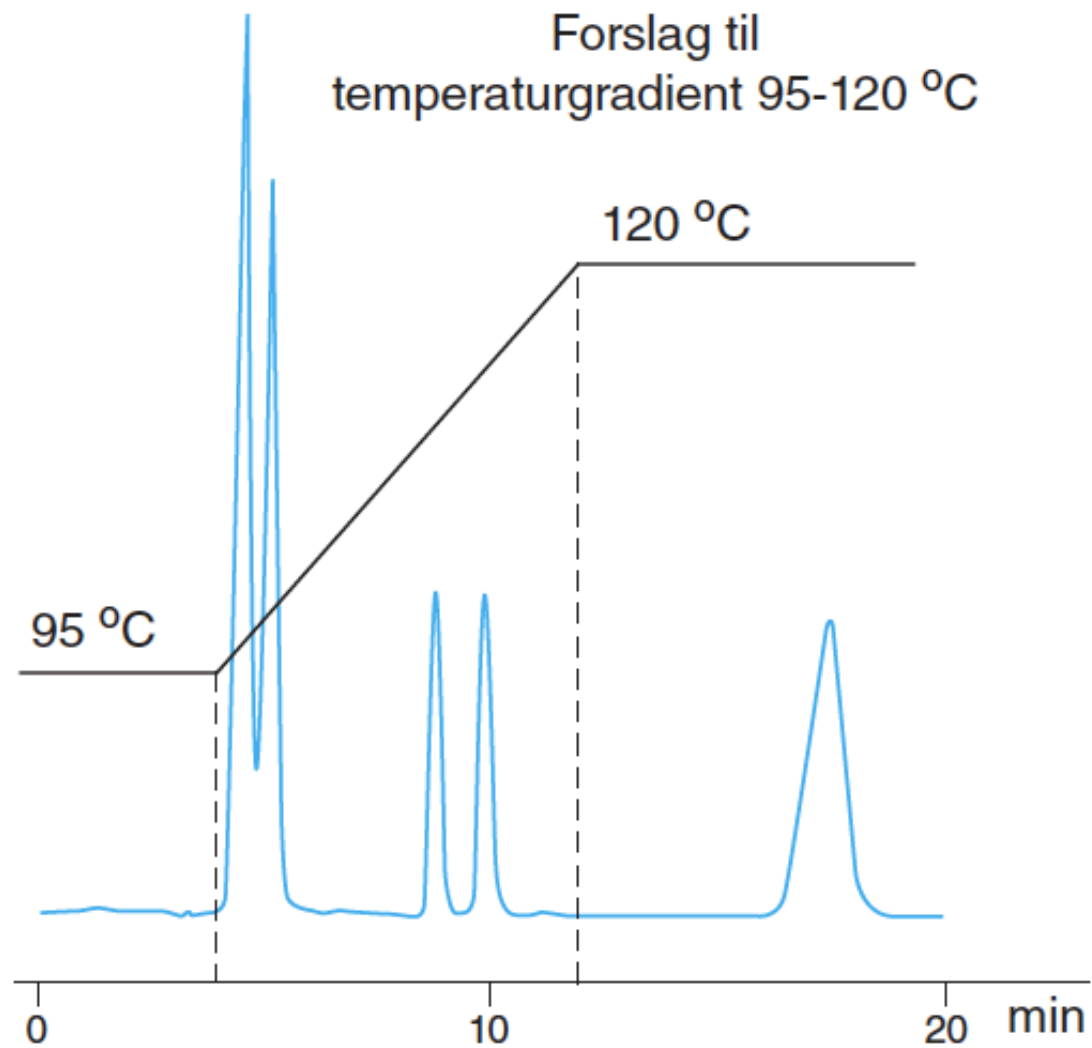
Figur 10.15. Topadskillelse, for lav temperatur.



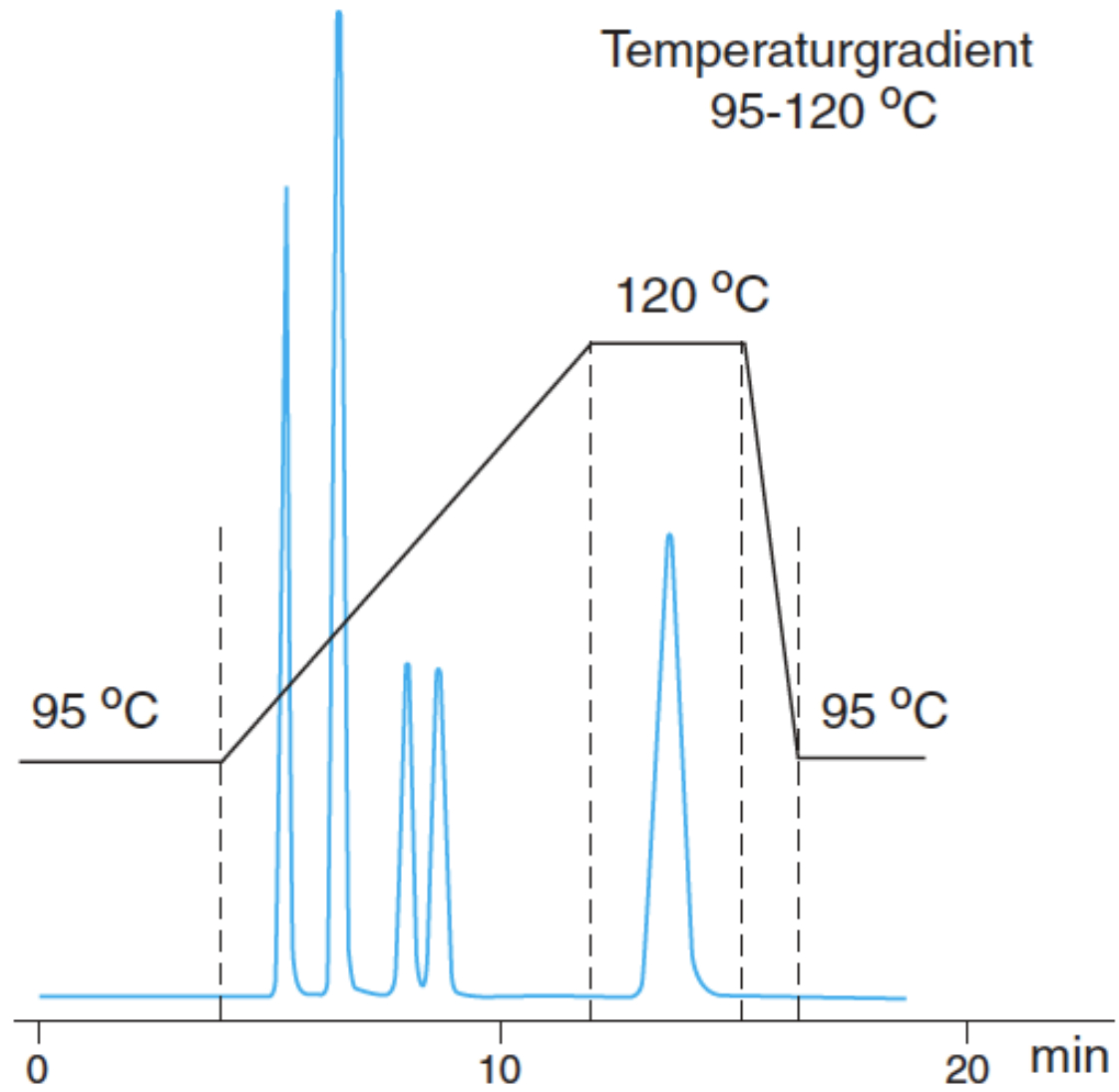
Figur 10.16. Isotermisk chromatogram med separationsproblem.



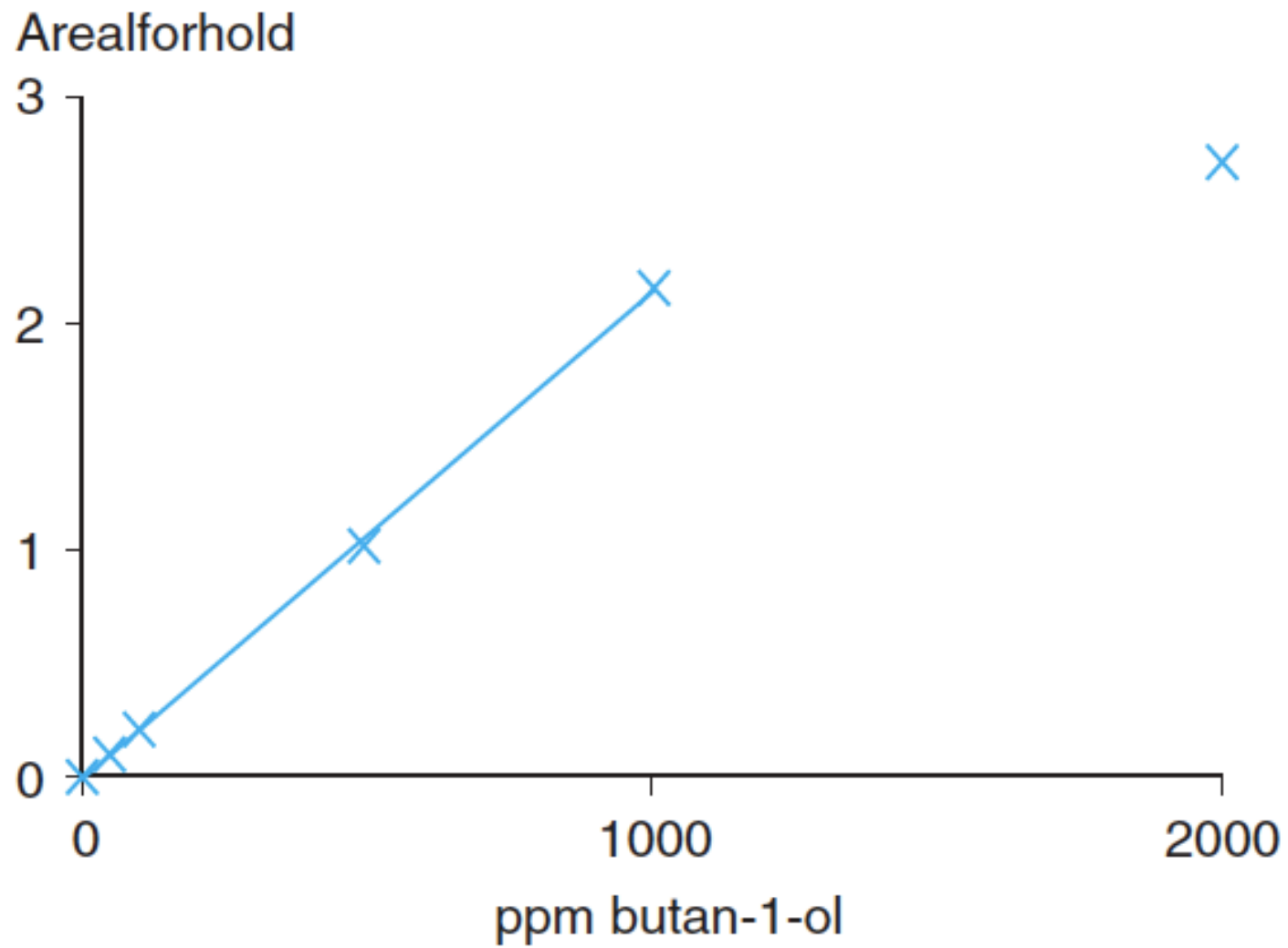
Figur 10.17. Temperaturprogram.



Figur 10.18. Forslag til temperaturprogram indtegnet på chromatogrammet fra figur 10.16.

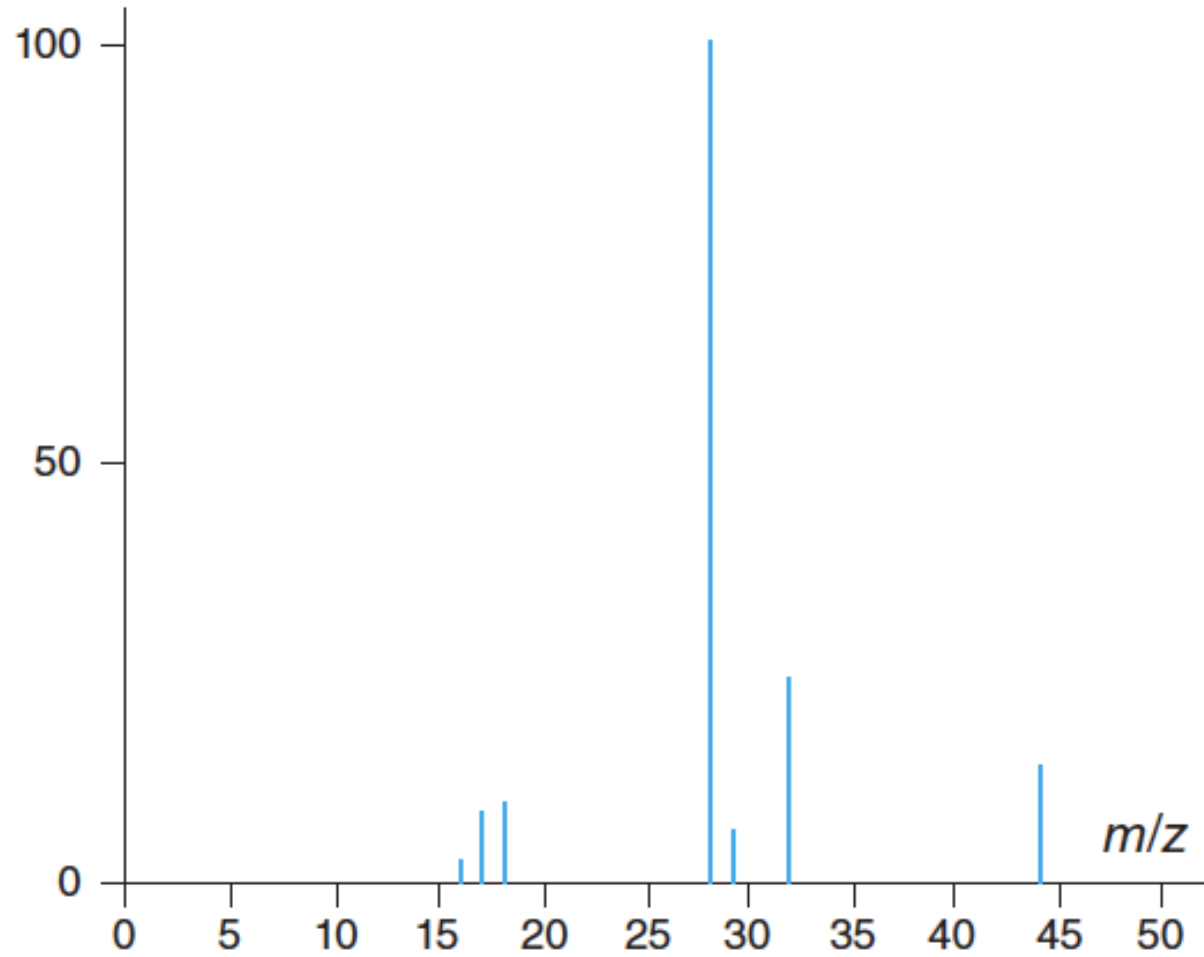


Figur 10.19. Chromatogram efter afvikling af temperaturprogram.

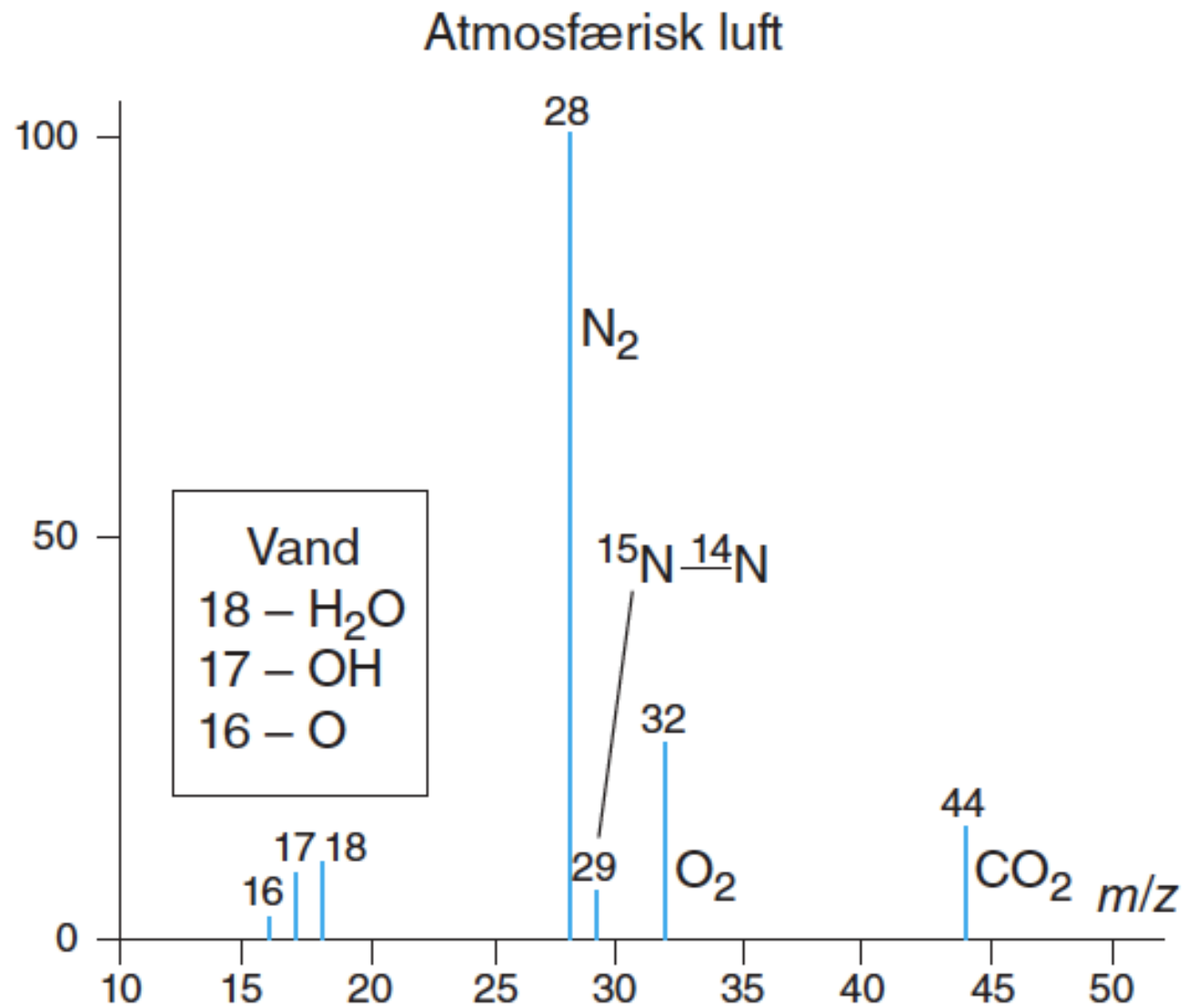


Figur 10.20. Linearitetsbestemmelse, GC.

Massespektrum



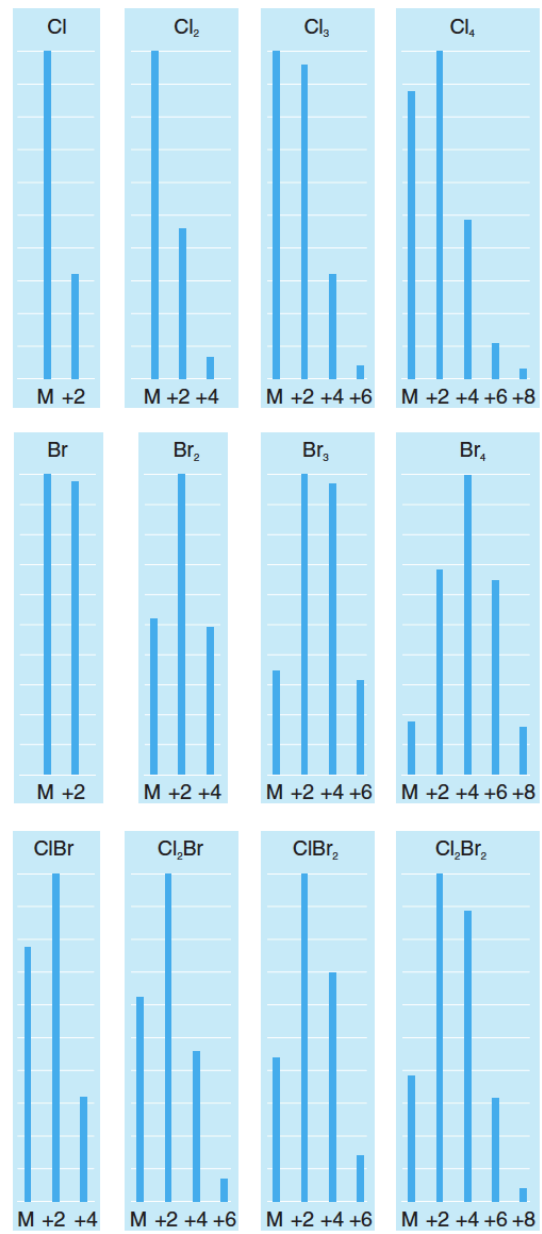
Figur 11.1. Eksempel på massespektrum.



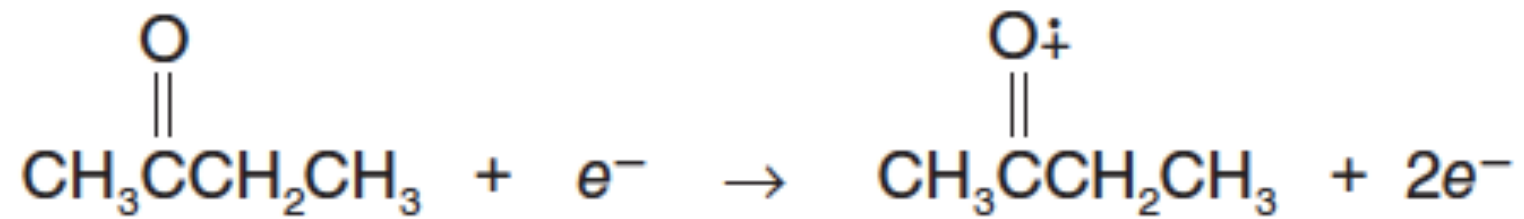
Figur 11.2. MS-spektrum, atmosfærisk luft.

<i>Relativ hyppighed</i>			
xM	xM	^{x+1}M	^{x+2}M
1H	100	-	-
^{12}C	100	1,1	-
^{14}N	100	0,4	-
^{16}O	100	-	0,2
^{32}S	100	0,8	4,4
^{35}Cl	100	-	32,5
^{79}Br	100	-	97,3

Tabel 11.1.



Figur 11.3. Isotopmønstre med Cl og Br.



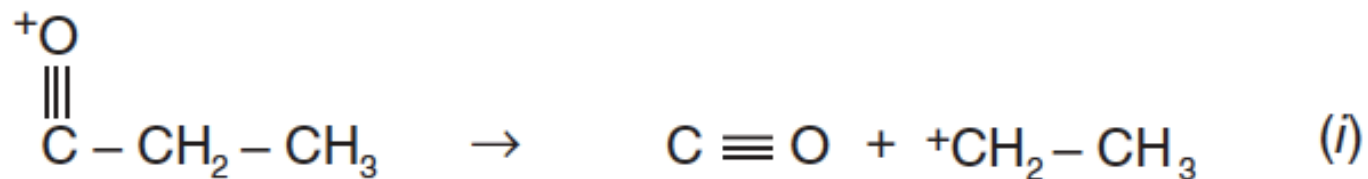
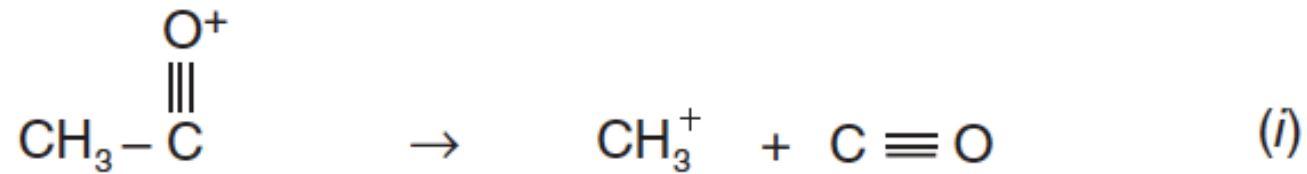
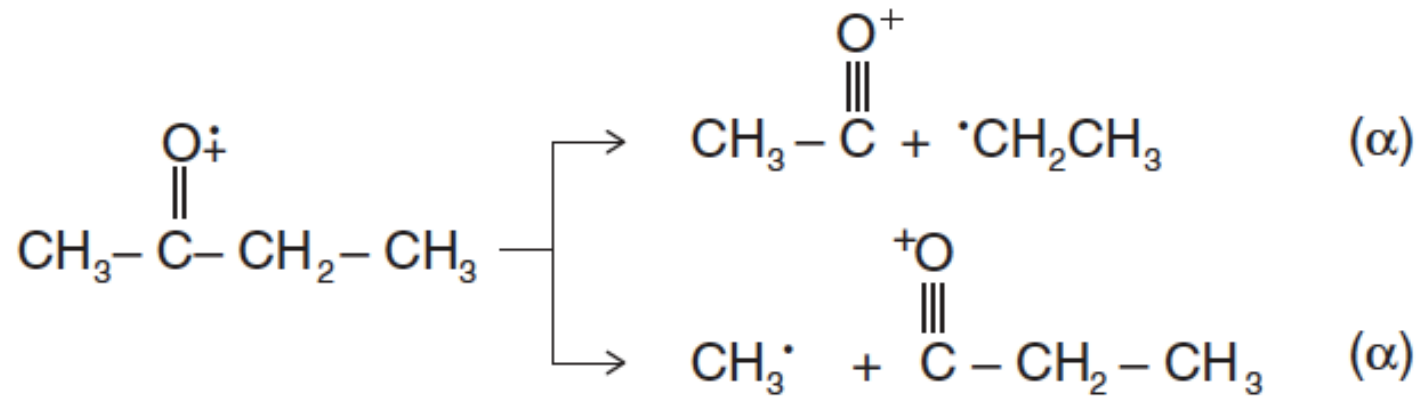
Figur 11.4. Dannelse af molekylærionen for butan-2-on

<i>Alkaner</i>	<i>m/z</i>
+CH ₃	15
+CH ₂ CH ₃	29
+CH ₂ CH ₂ CH ₃	43
+CH ₂ CH ₂ CH ₂ CH ₃	57
<i>Alkoholer</i>	
+OH	17
+CH ₂ OH	31
+CHOHCH ₃	45
+CHOHCH ₂ CH ₃	59
+CHOHCH ₂ CH ₂ CH ₃	73
<i>Aminer</i>	
+CH ₂ NH ₂	30
+CH ₂ NHCH ₃	44
+CH ₂ NHCH ₂ CH ₃	58
+CH ₂ NHCH ₂ CH ₂ CH ₃	72

Tabel 11.2.

alkaner	15, 29, 43, 57, 71, 85, 99
alkener ⁹¹	27, 41, 55, 69, 83, 97
alkoholer ⁹²	17, 31, 45, 59, 73, 87, 101
Aromater	38, 39, 50-52, 63-65, 75-78
syrer/estere	45, 59, 73, 87, 101
alkylaminer	30, 44, 58, 72, 86, 100
chloralkyler	49, 63, 77, 91, 105
alkylsilaner ⁹³	31, 45, 59, 73, 87, 101

Tabel 11.3.

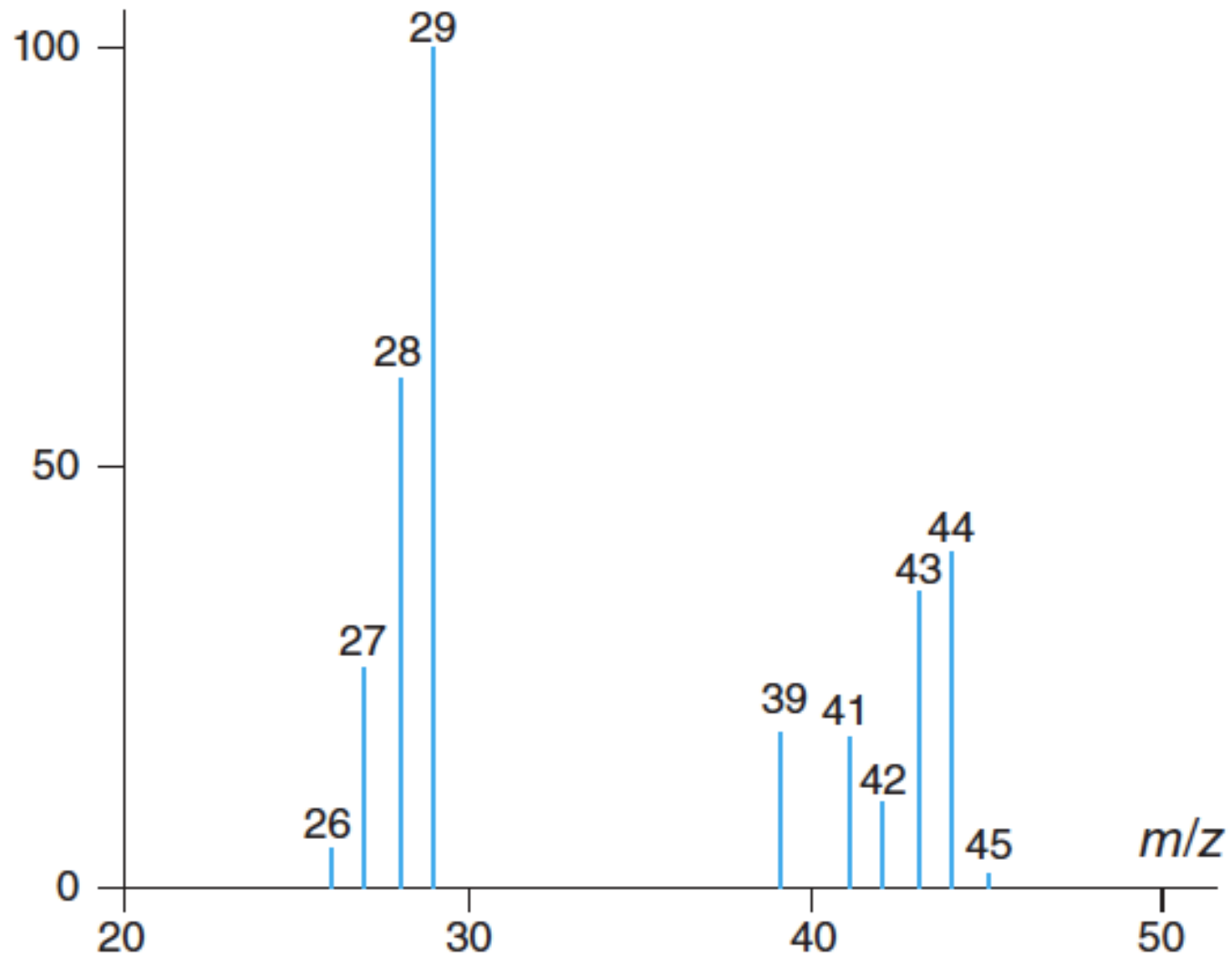


Figur 11.5. Videre fragmentering af butan-2-on, øverst α -fragmenteringer af molekylarionen, nederst nye fragmenter dannes ved i fragmenteringer.

<i>Tabtfragment</i>	<i>m</i>
H ₂ O	18
CO og C ₂ H ₂	28
CH ₂ O	30
CH ₃ + H ₂ O	33
CH ₃ CH ₂ OH	46

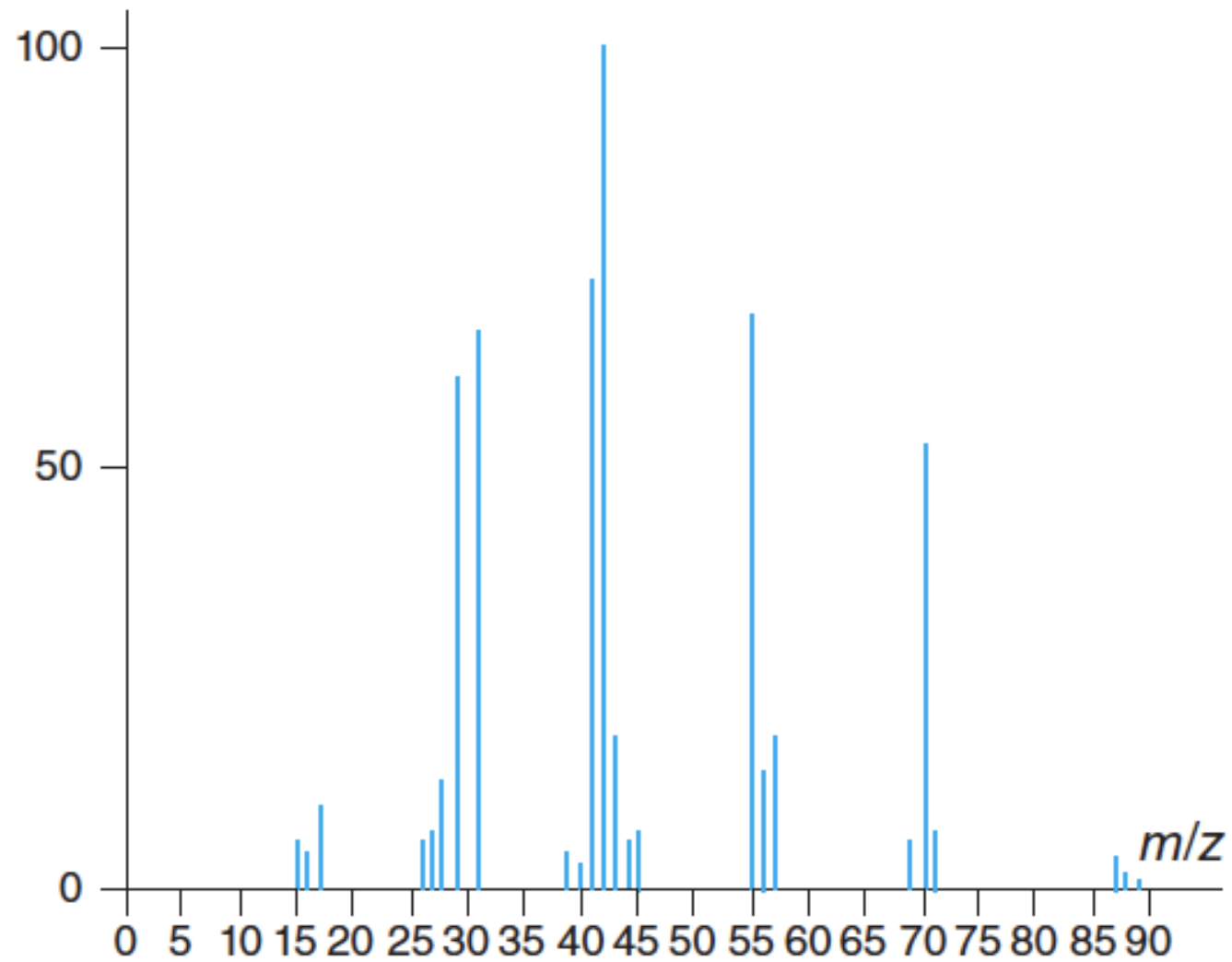
Tabel 11.4.

Massespektrum



Figur 11.6. MS-spektrum til tydning.

Massespektrum

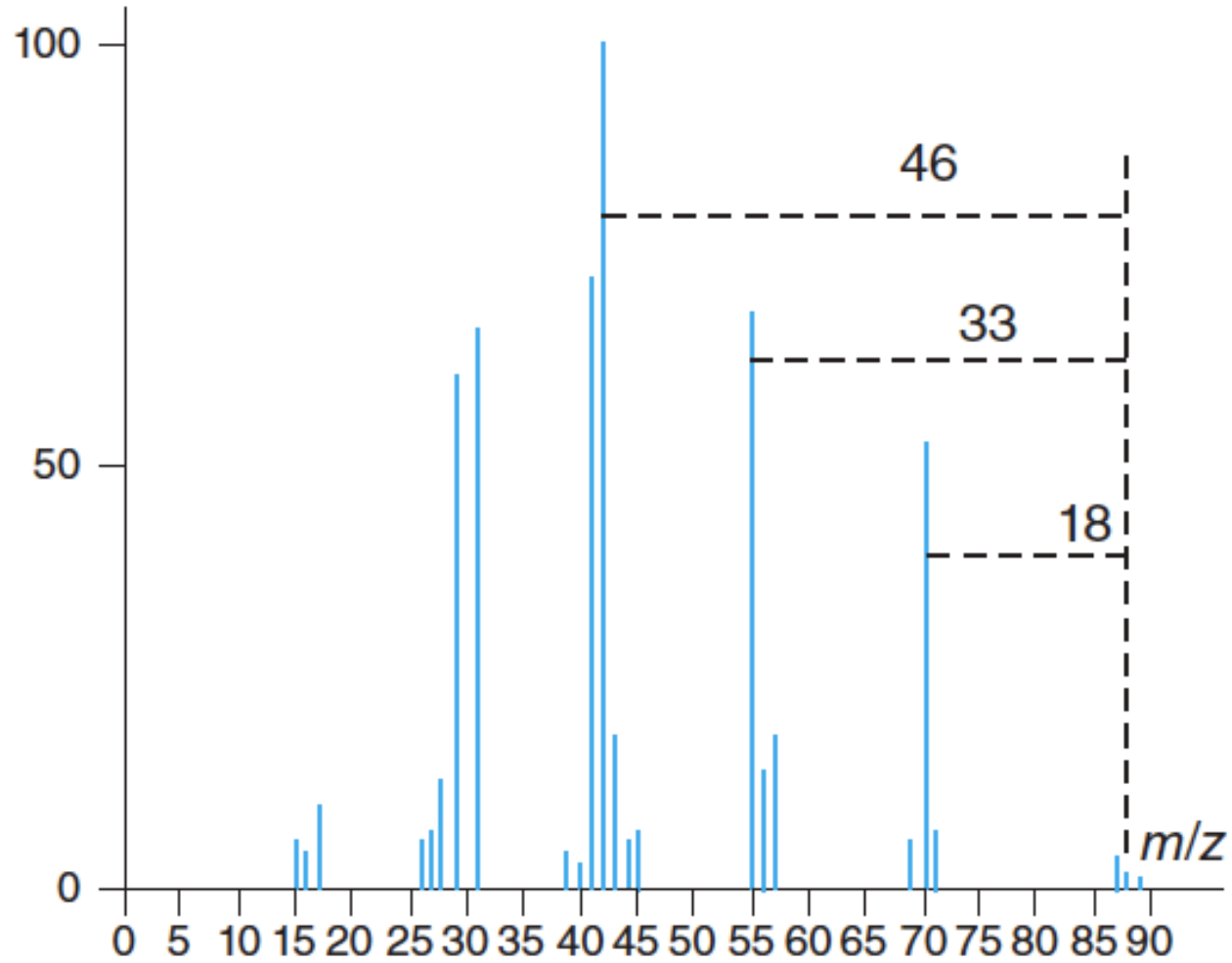


Figur 11.7. MS-spektrum til tydning.

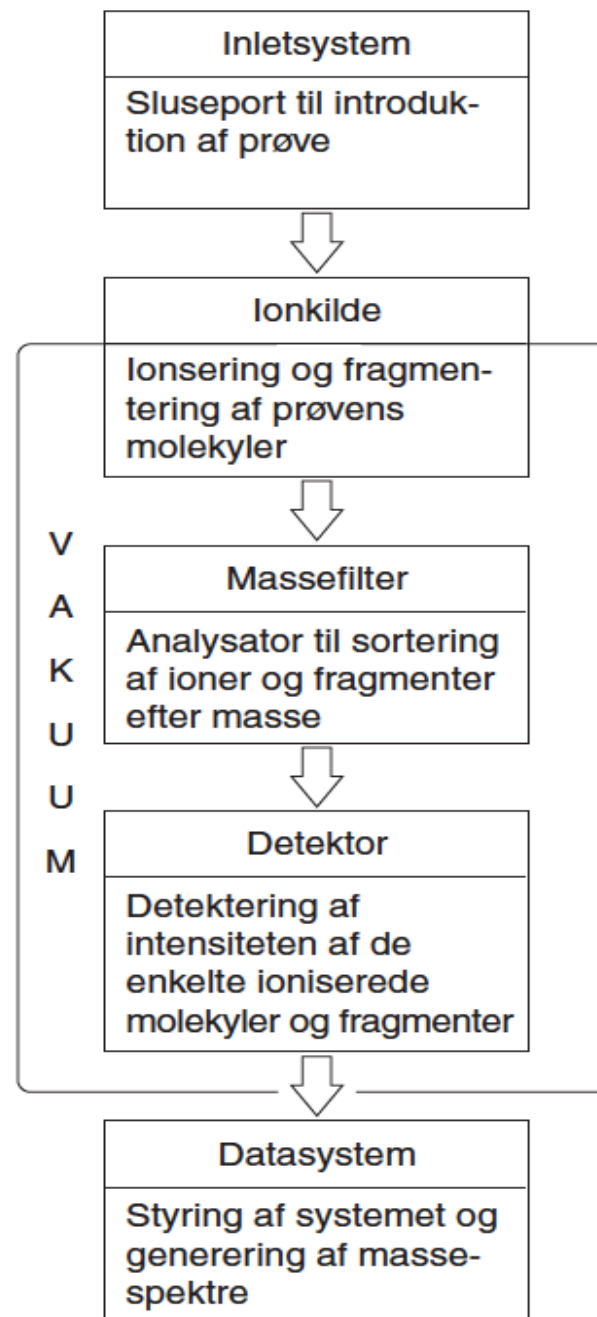
alkan	15, 29, 43, 57, 71, 85, 99
alkohol	17, 31, 45, 59, 73, 87, 101
spektrum	15, 16, 17, 26, 27, 28, 29, 31, 39, 40, 41, 42, 43, 44, 45, 55, 56, 57, 69, 70, 71, 87, 88, 89

Tabel 11.3 i uddrag.

Massespektrum

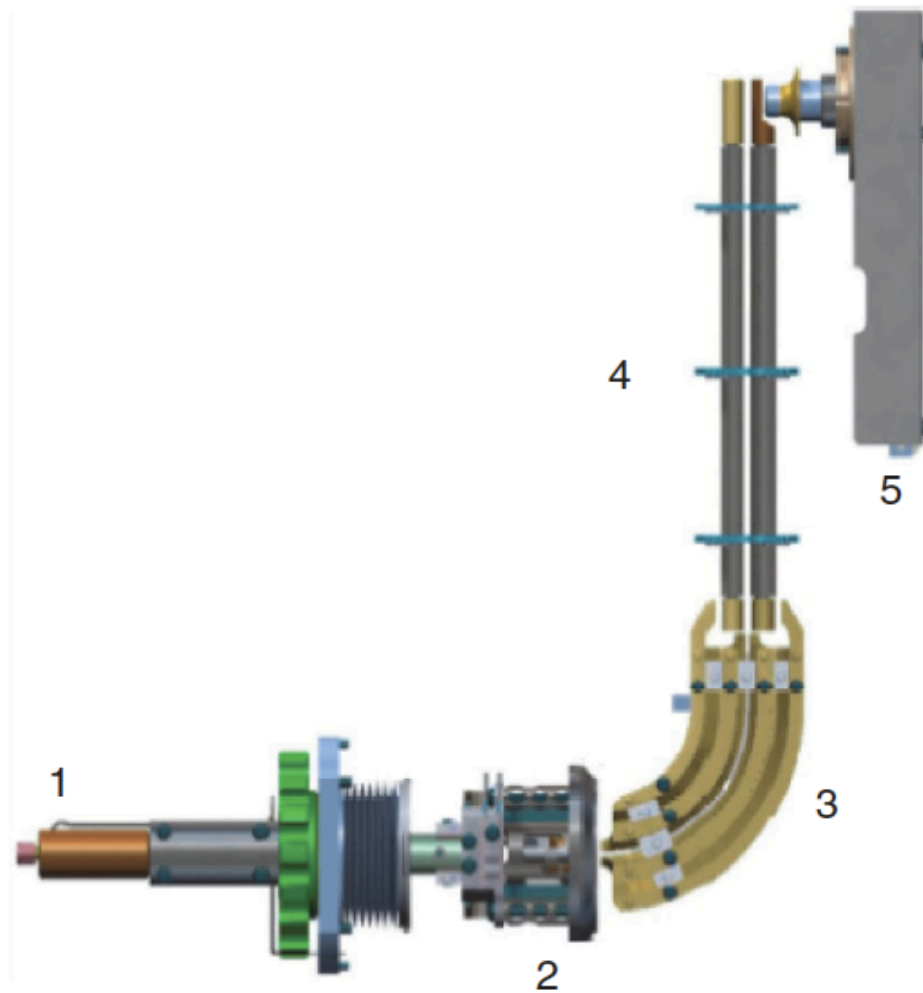


Figur 11.8. MS-spektrum, fragmentab.

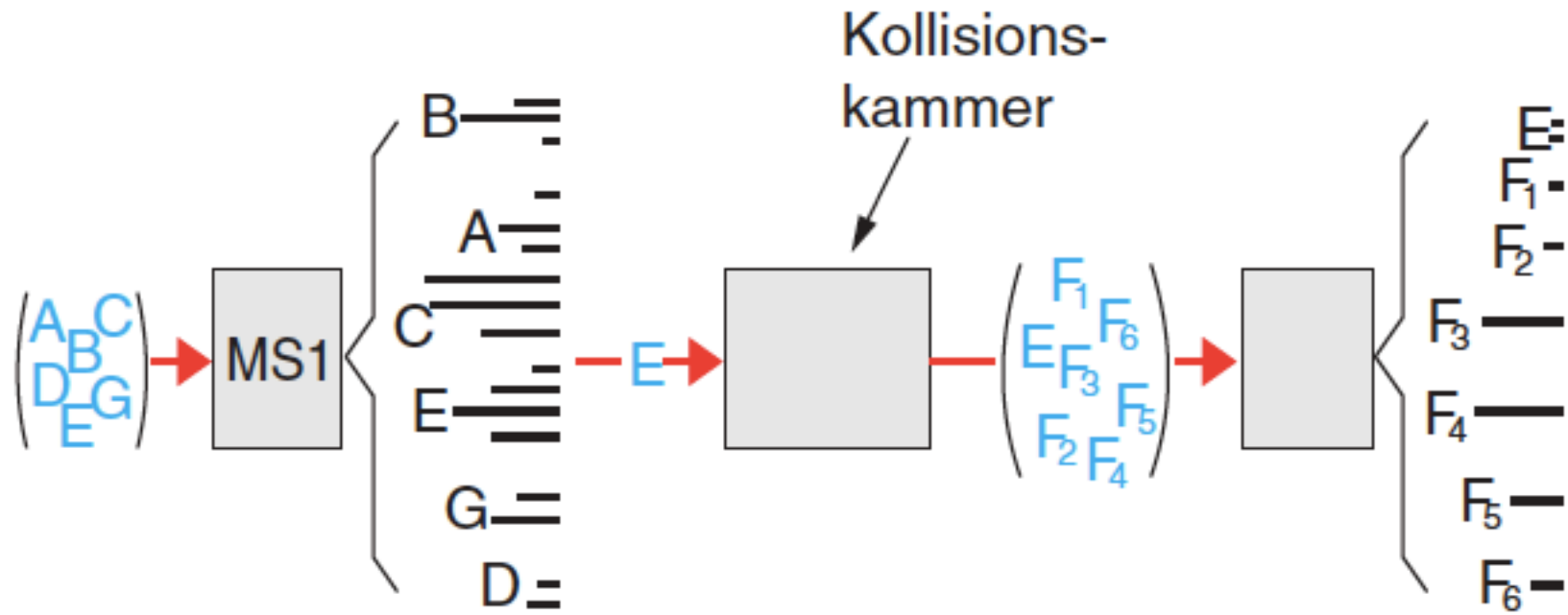


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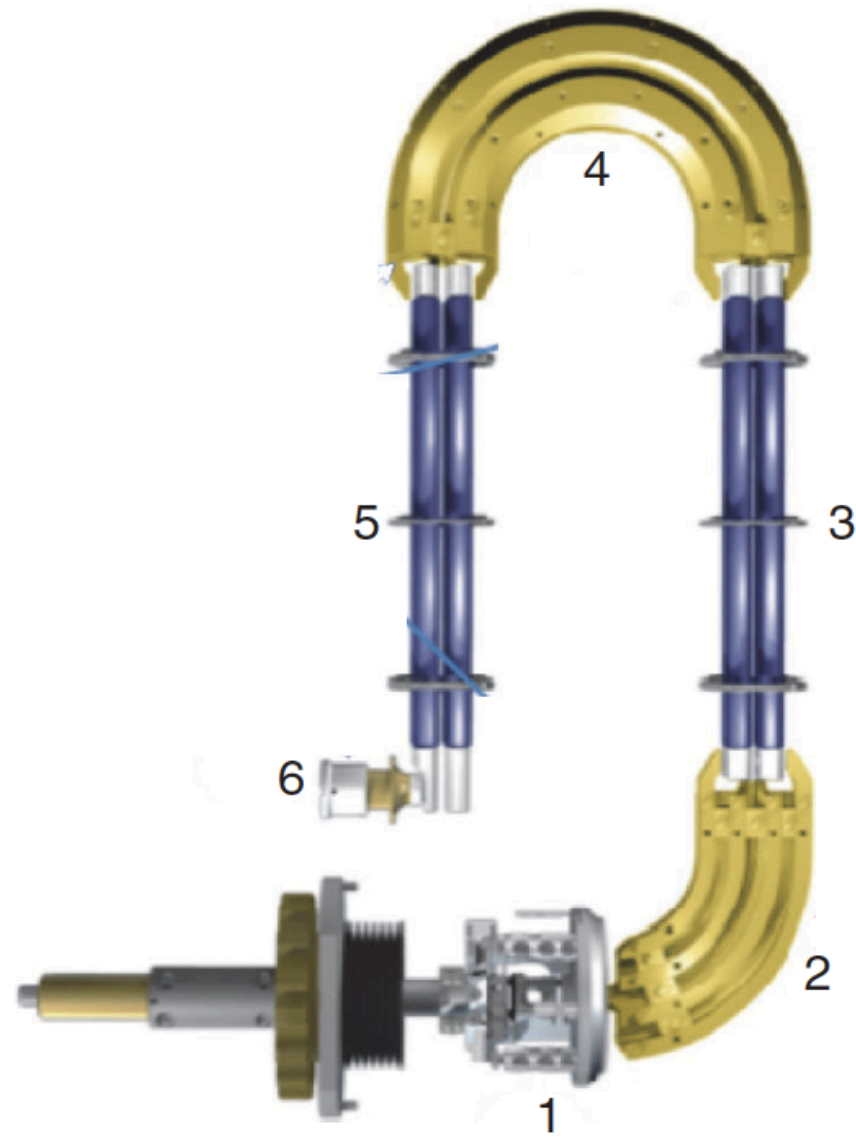
Figur 11.9. Massespektrometrets opbygning.



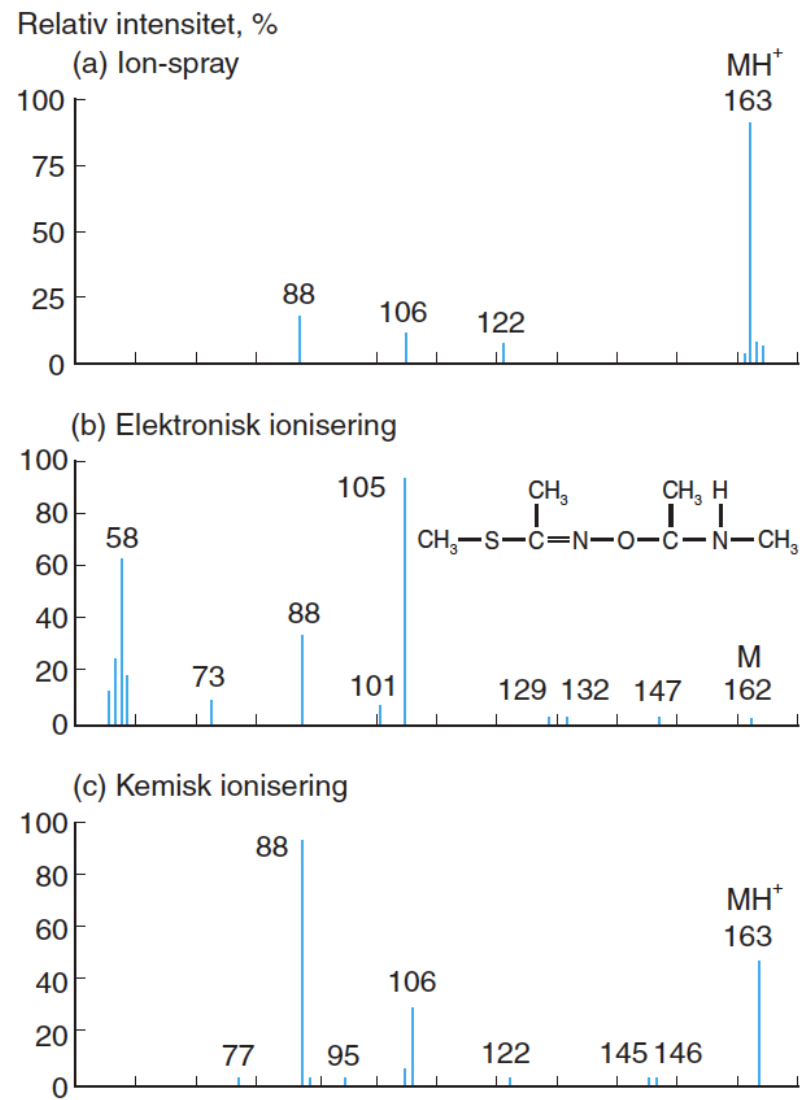
Figur 11.10. (Bruker) eksempel på instrumentopbygning: Til venstre ses interface fra GC (1), ionisering (2), fokusering og rensning af ionstrålen (3), massefilteret, her en quadropol (4) og detektoren (5).



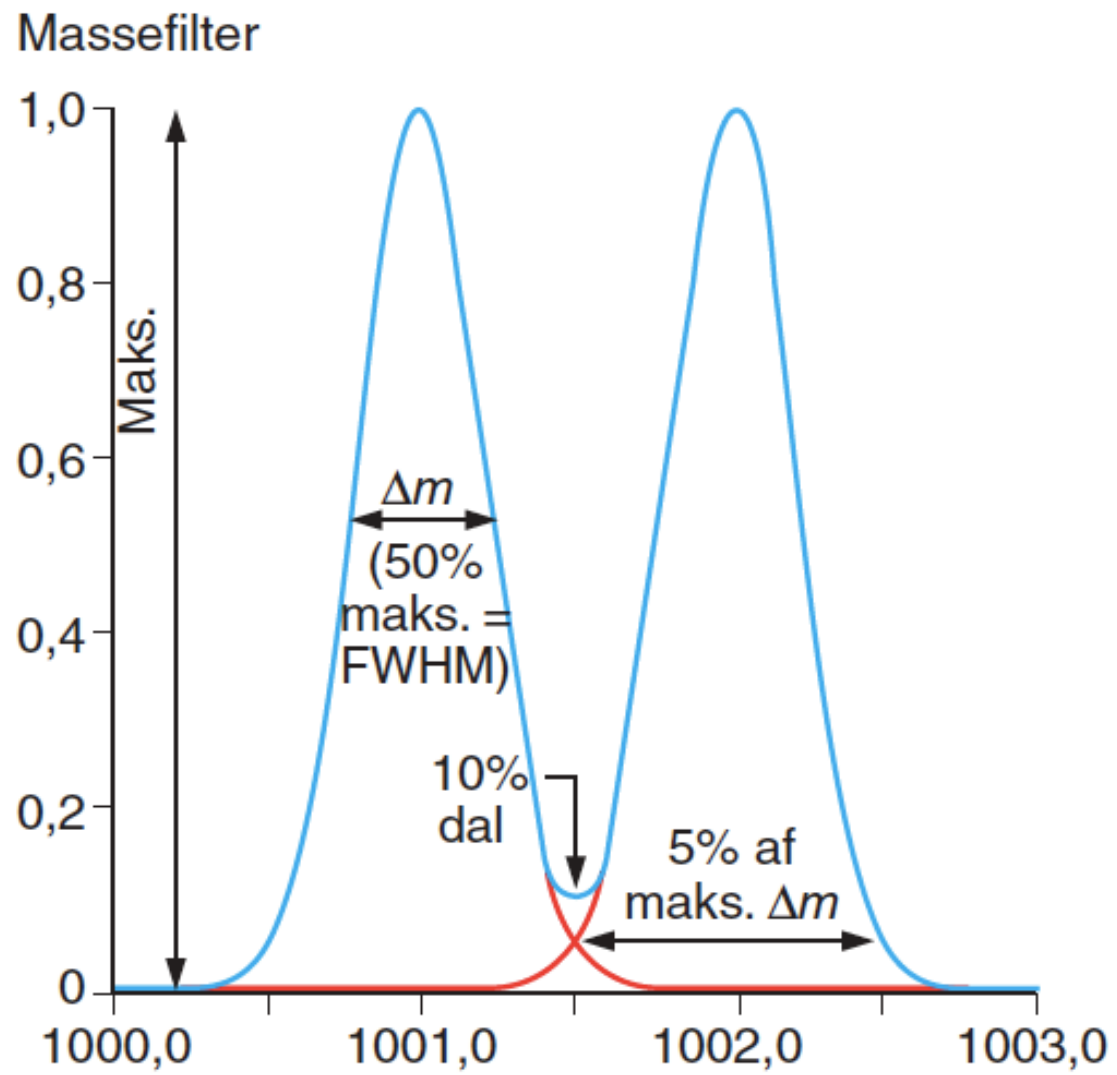
Figur 11.11. Tandem-MS



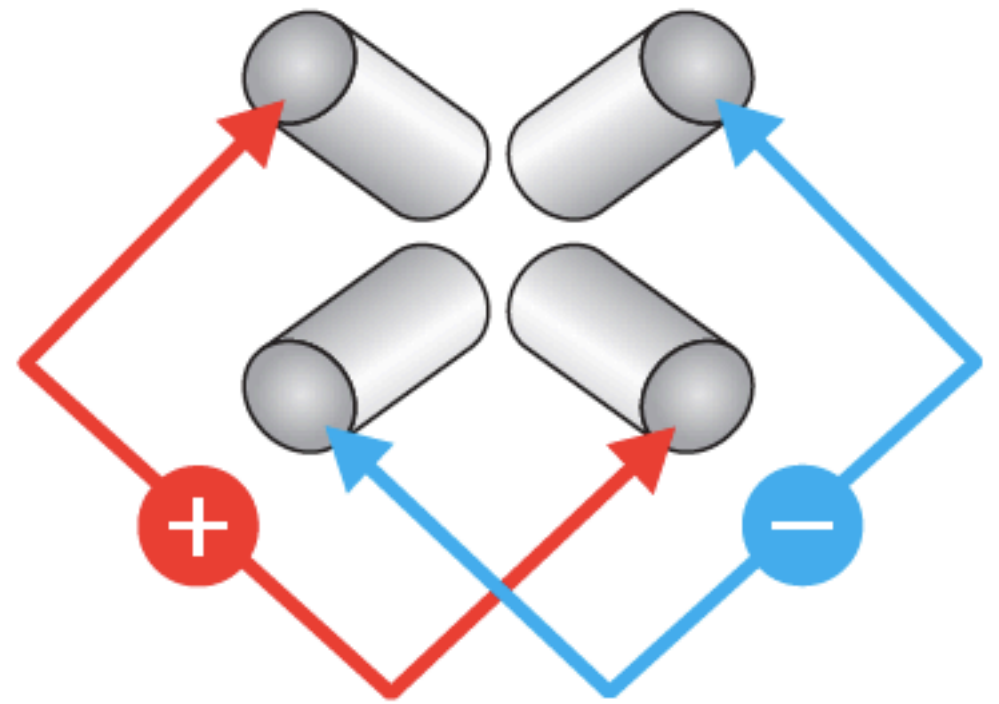
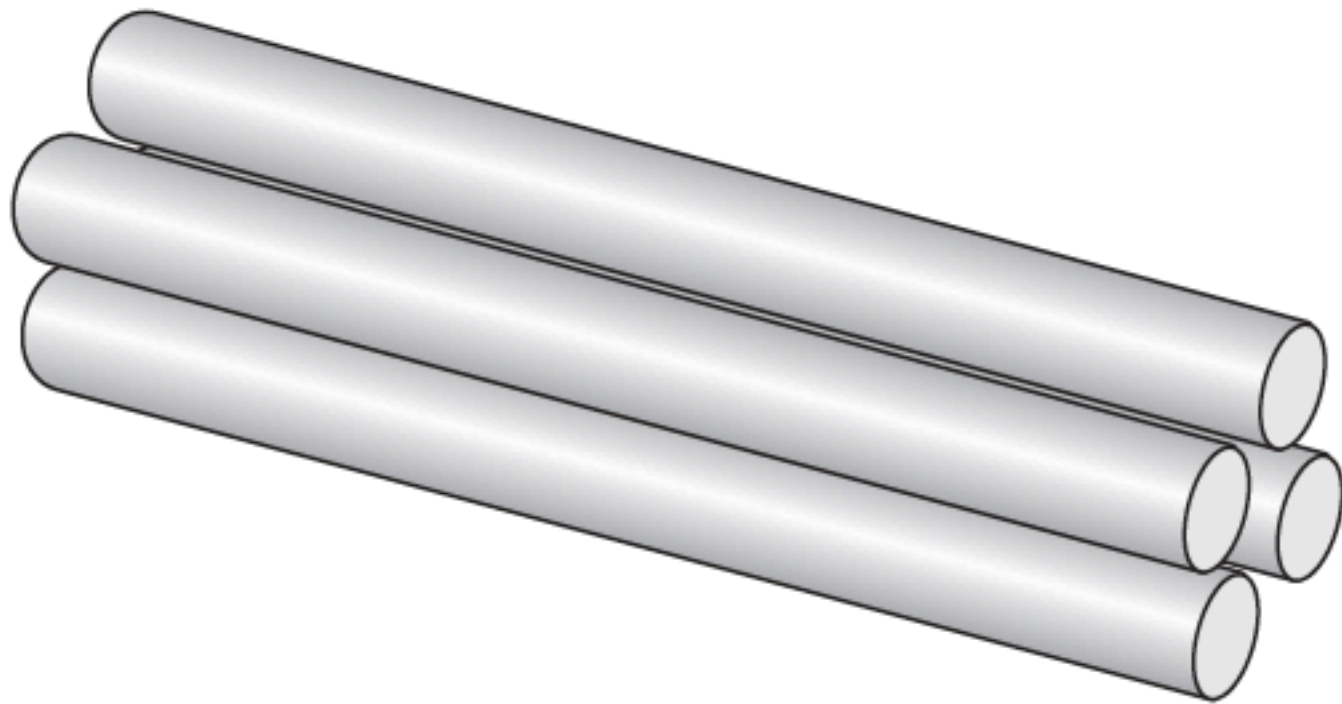
Figur 11.12. Trippelquad eller QqQ.



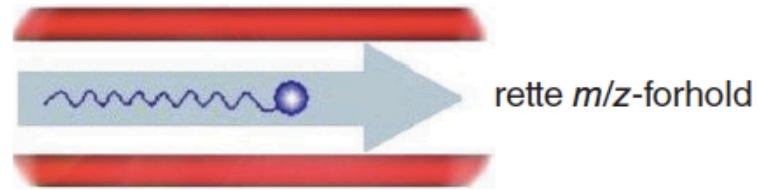
Figur 11.13. Prøven i alle tre spektre er Methomyl (et carbamat pesticid). Se strukturen i spektrum b). a) ESI, b) EI og c) CI.



Figur 11.14. Masseresolution
(FWHM = Full Width at Half Maximum)



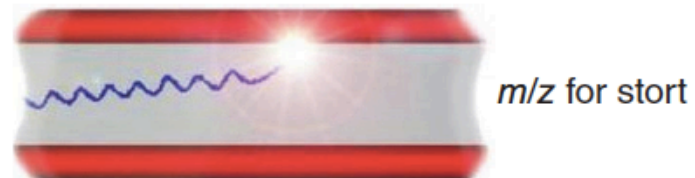
Figur 11.15. Quadropol



- A) Kun ioner med rette m/z -forhold er stabile flyvere i quadropolens elektriske felt og kommer igennem til detektoren.

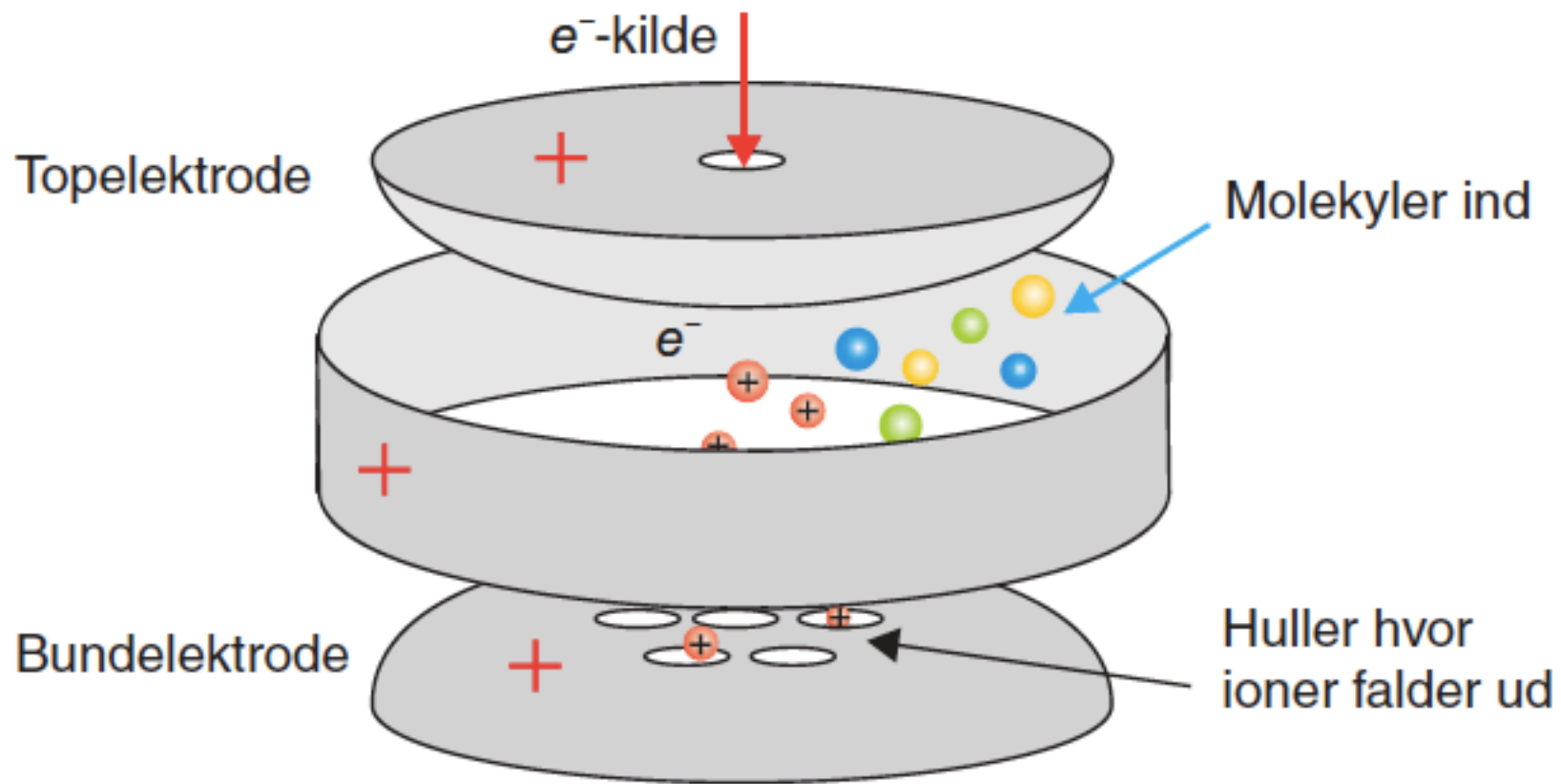


- B) Lette ioner vil blive mere påvirket af den skiftende RF-spænding, og kommer derved i svingninger, der bliver så store at de vil kollideres med en stav eller smutte ud mellem to stave og forsvinde.

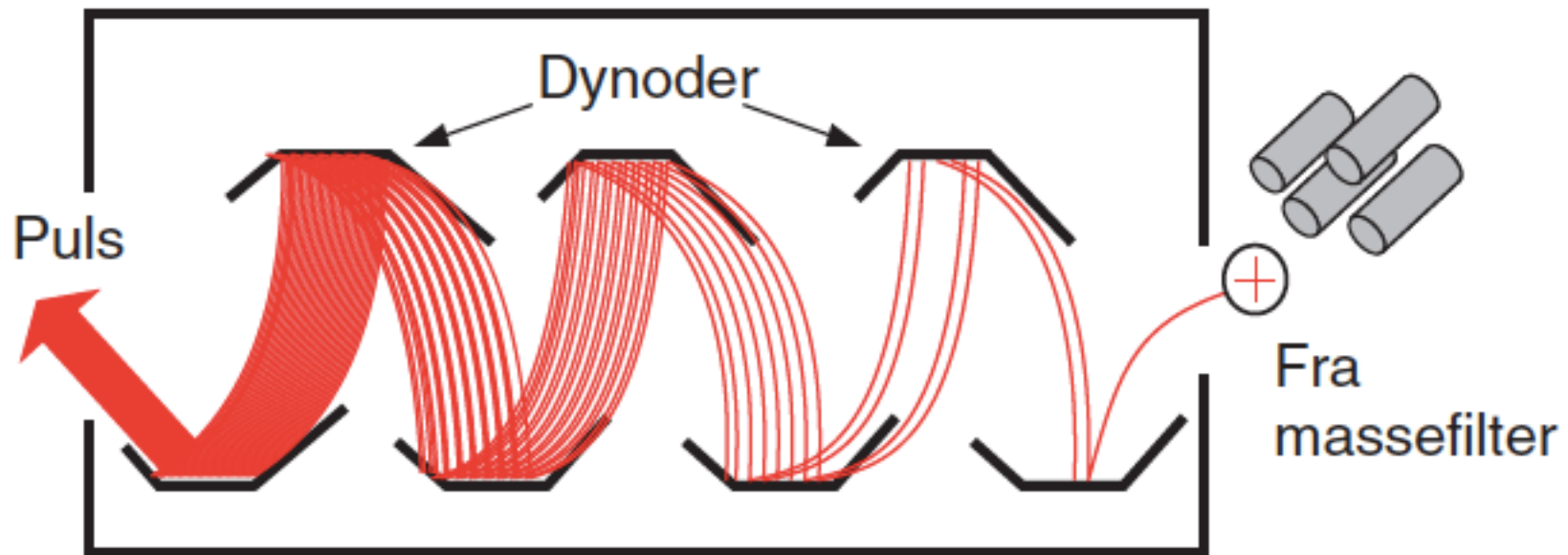


- C) Tunge ioner påvirkes ikke særligt af den skiftende RF-spænding, men vil gradvist drive i det konstante DC-felt. RF-feltet er ikke stærkt nok til at trække ionen tilbage, så den vil kollideres med en stav eller smutte ud mellem to stave og forsvinde.

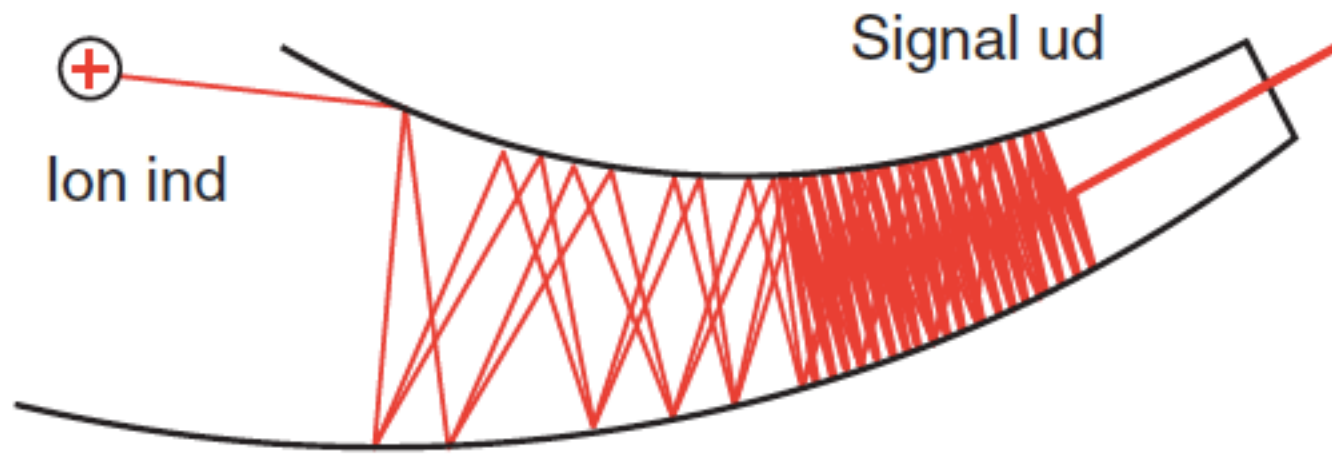
Figur 11.16. Funktion af quadropol.



Figur 11.17. Iontrap-massefilter.

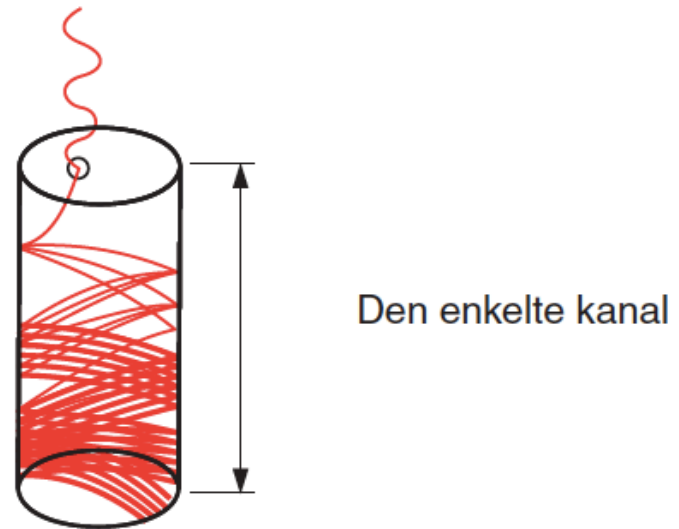
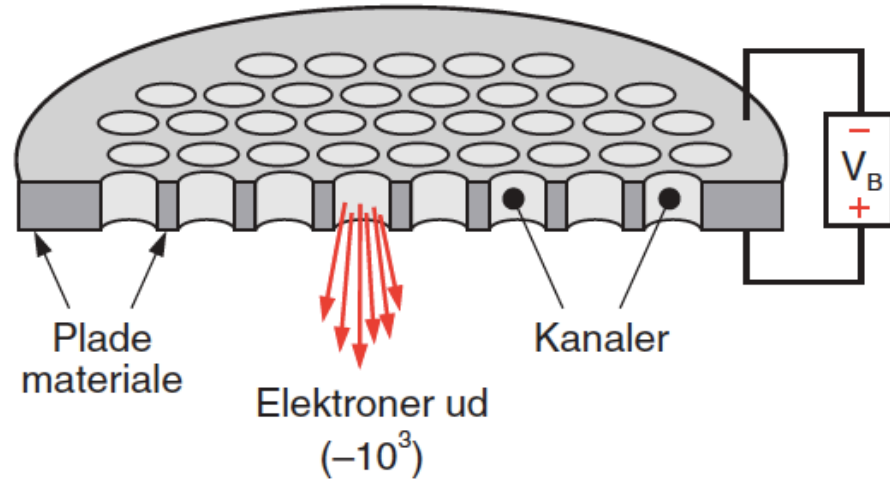


Figur 11.18. Elektronmultiplikator.

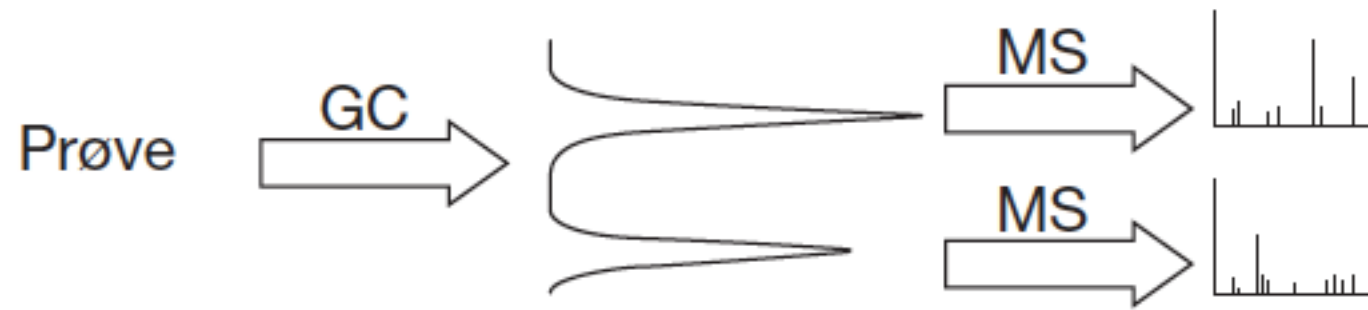


Figur 11.19. Elektronmultiplikator fra sektorinstrument.

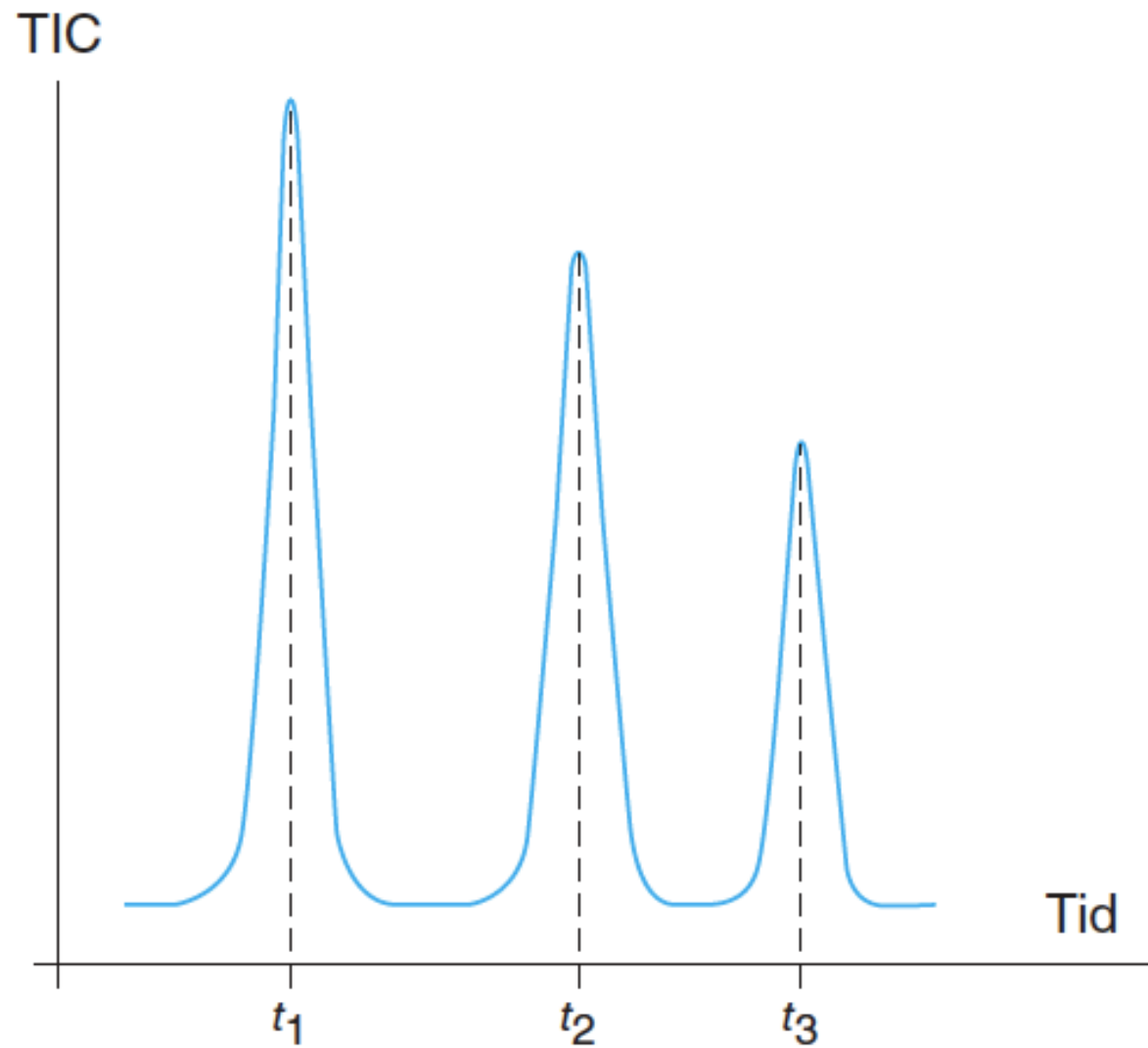
Hele pladen, ca. 50% af arealet er kanaler



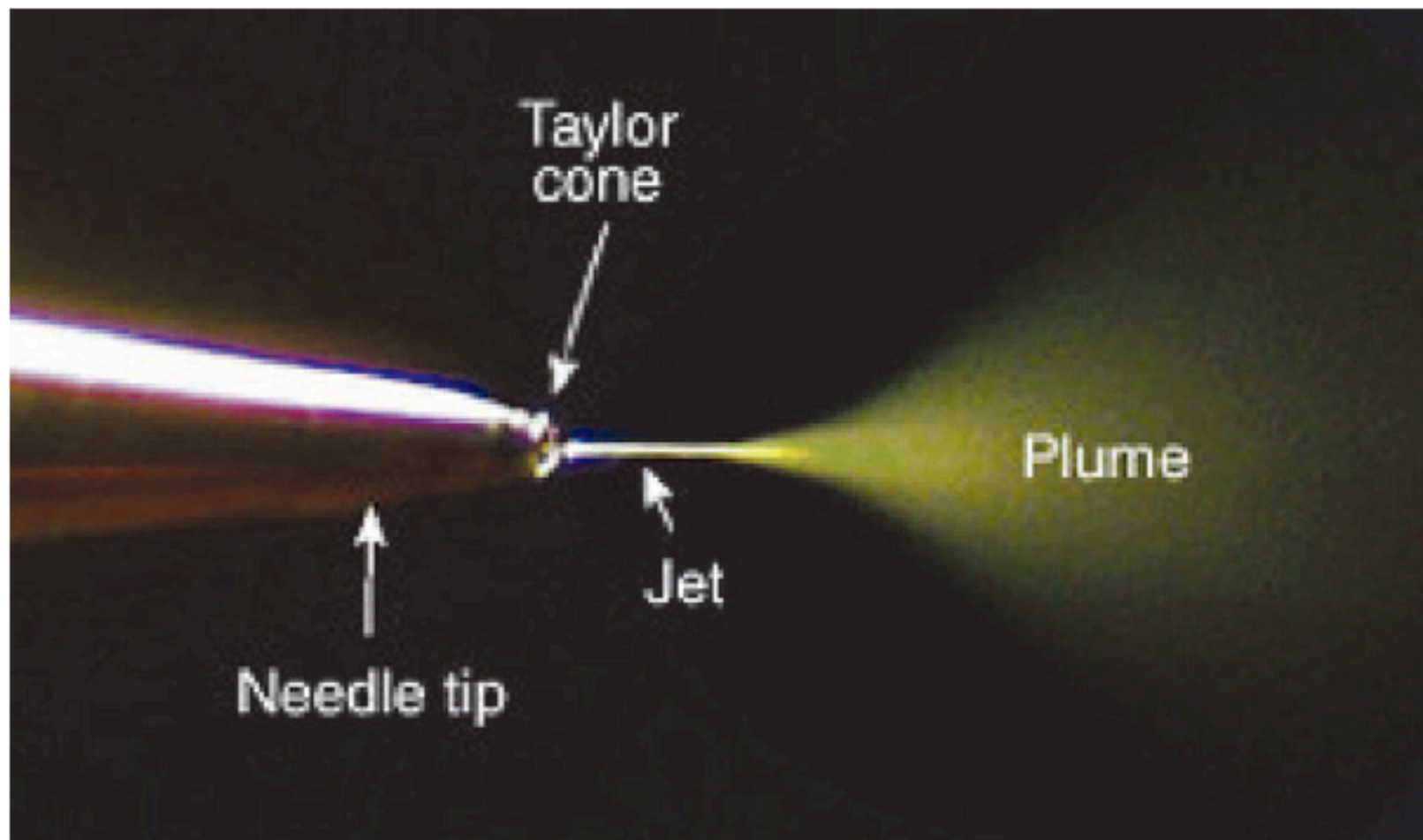
Figur 11.20. MicroChannel Plate



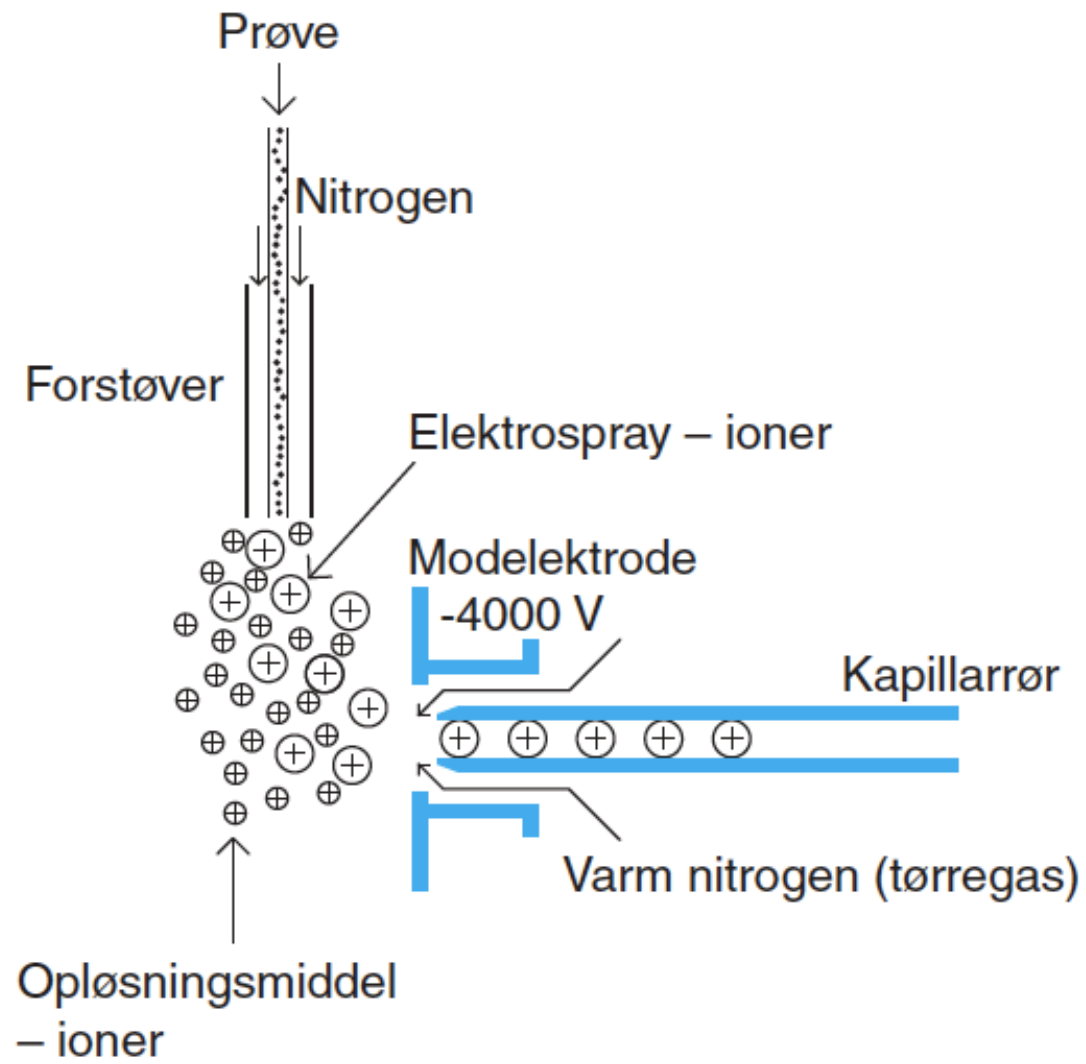
Figur 11.21. GC-MS princip.



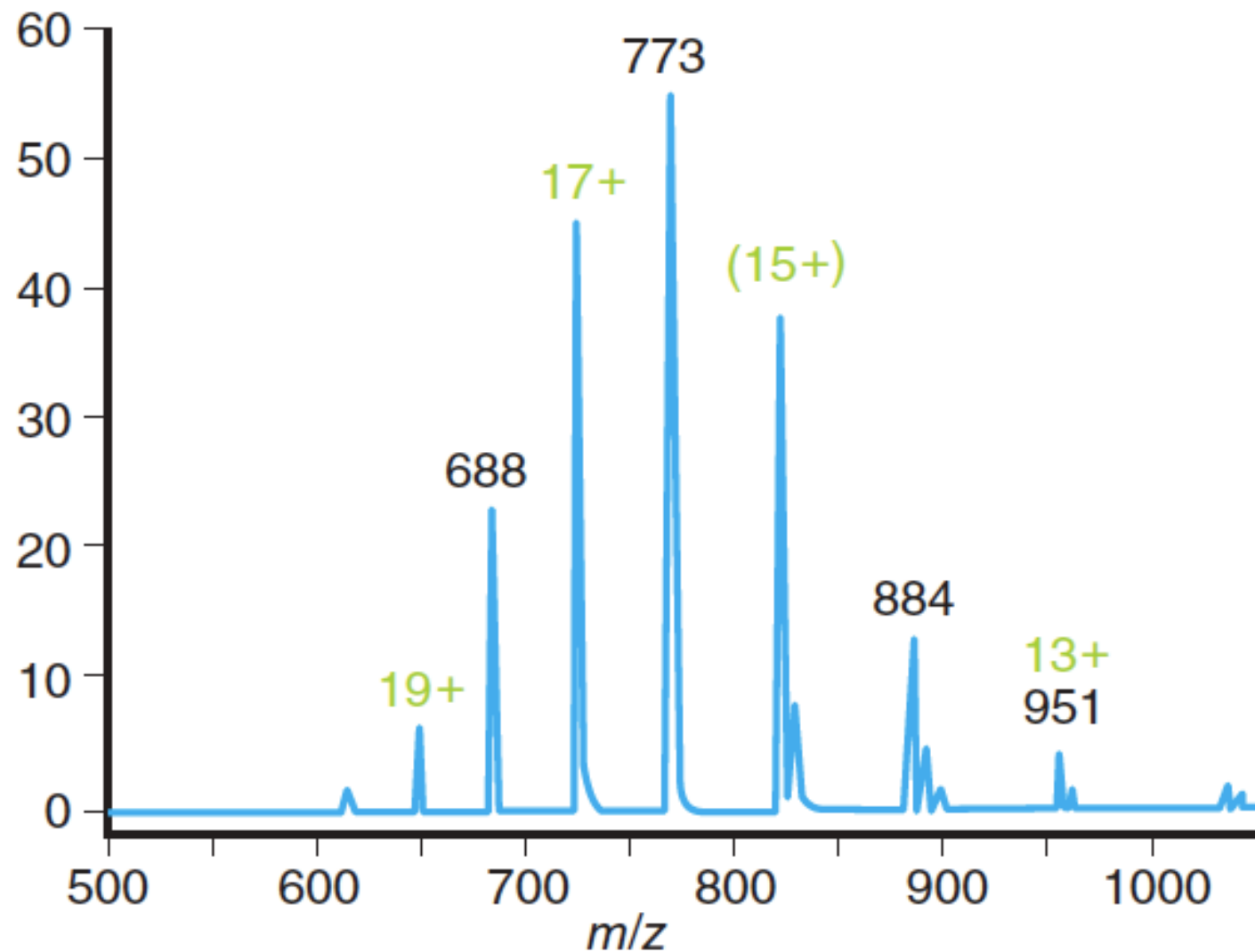
Figur 11.22. Hyppighed som funktion af tiden.



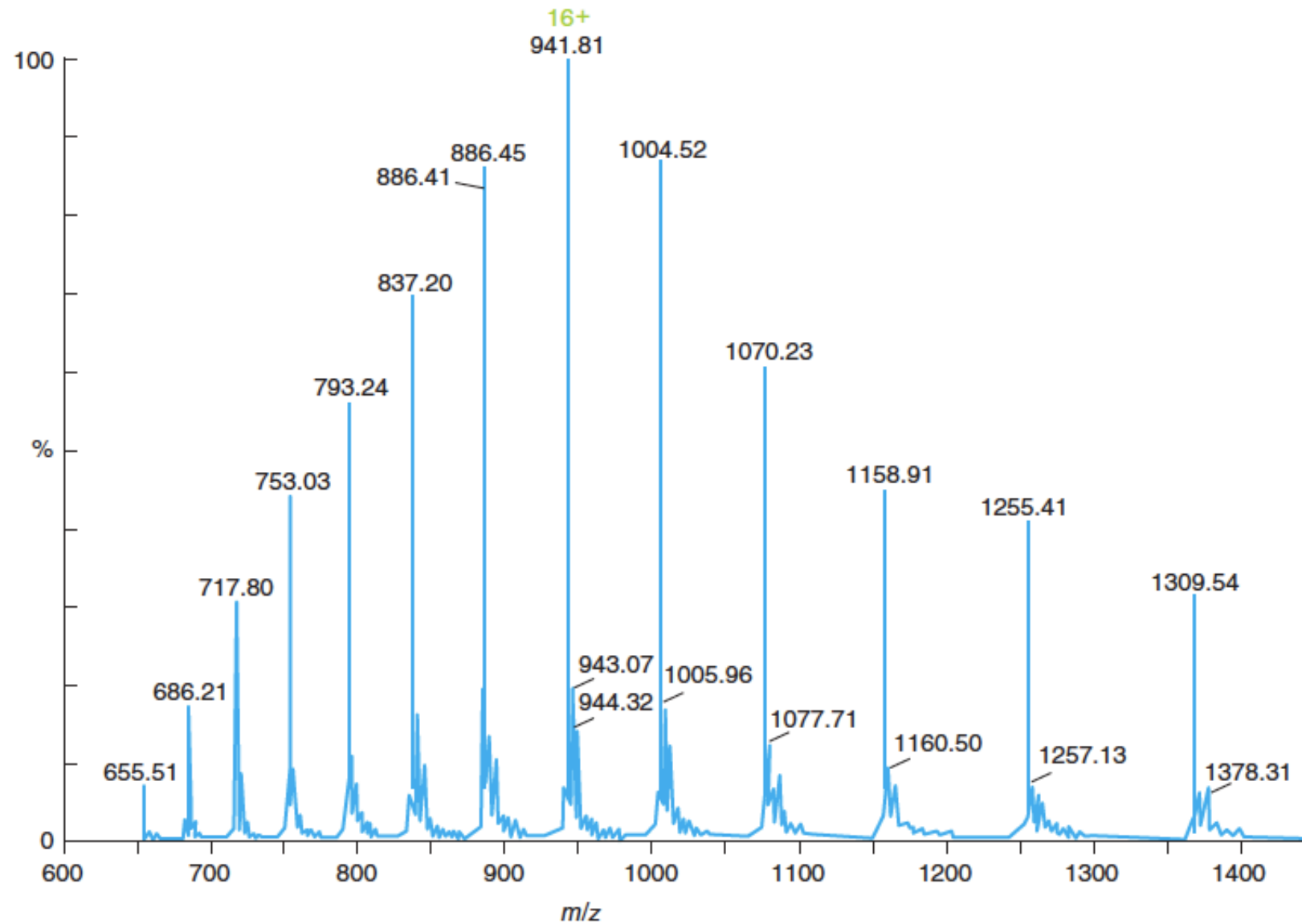
Figur 11.23. Foto af ESI (Aarhus Universitet).



Figur 11.24. Elektrospray-ionisering.

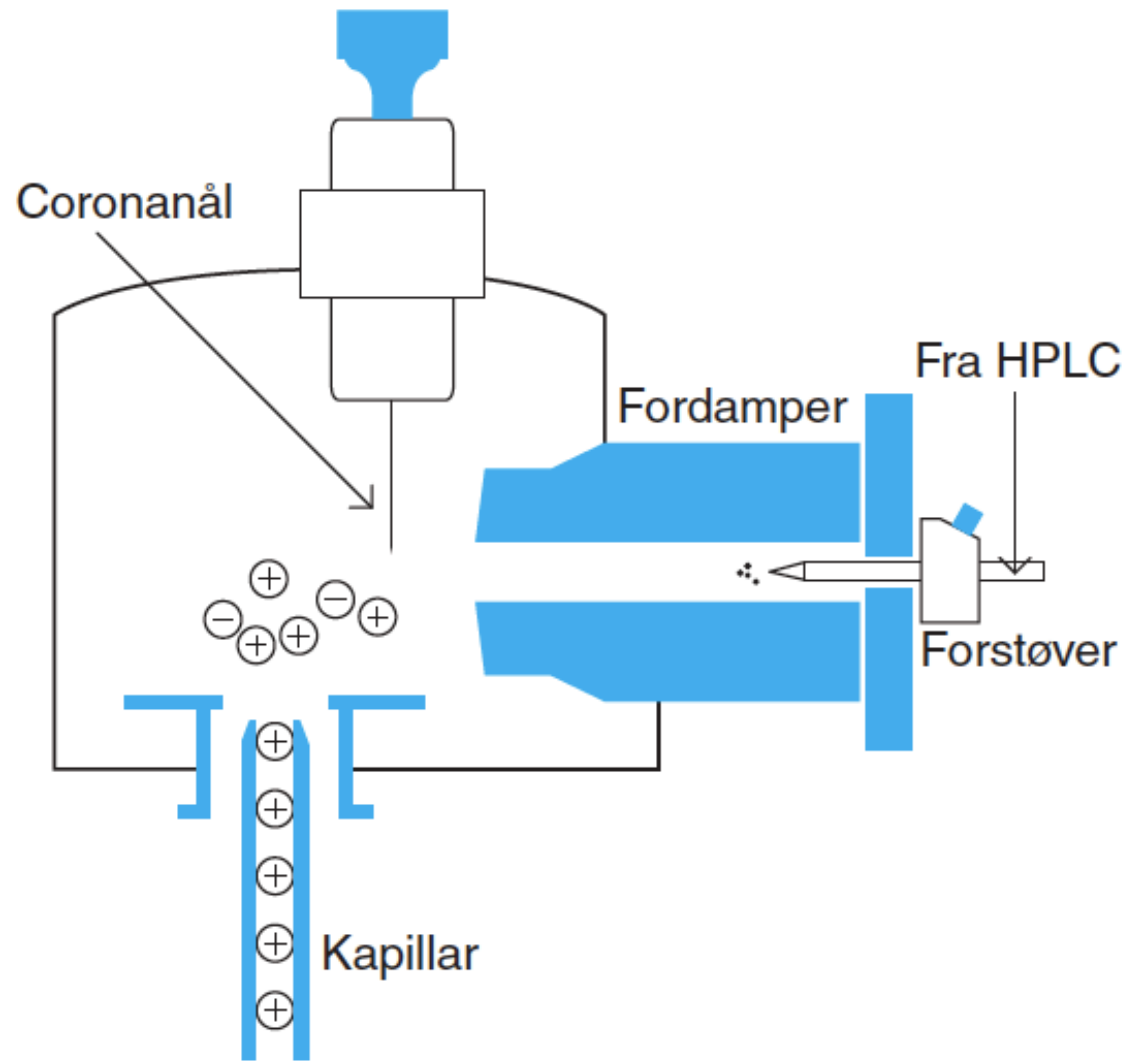


Figur 11.25. Eksempel på et ESI-spekter af cytochrome *c*, et protein ($1,5 \cdot 10^{-6}$ M).

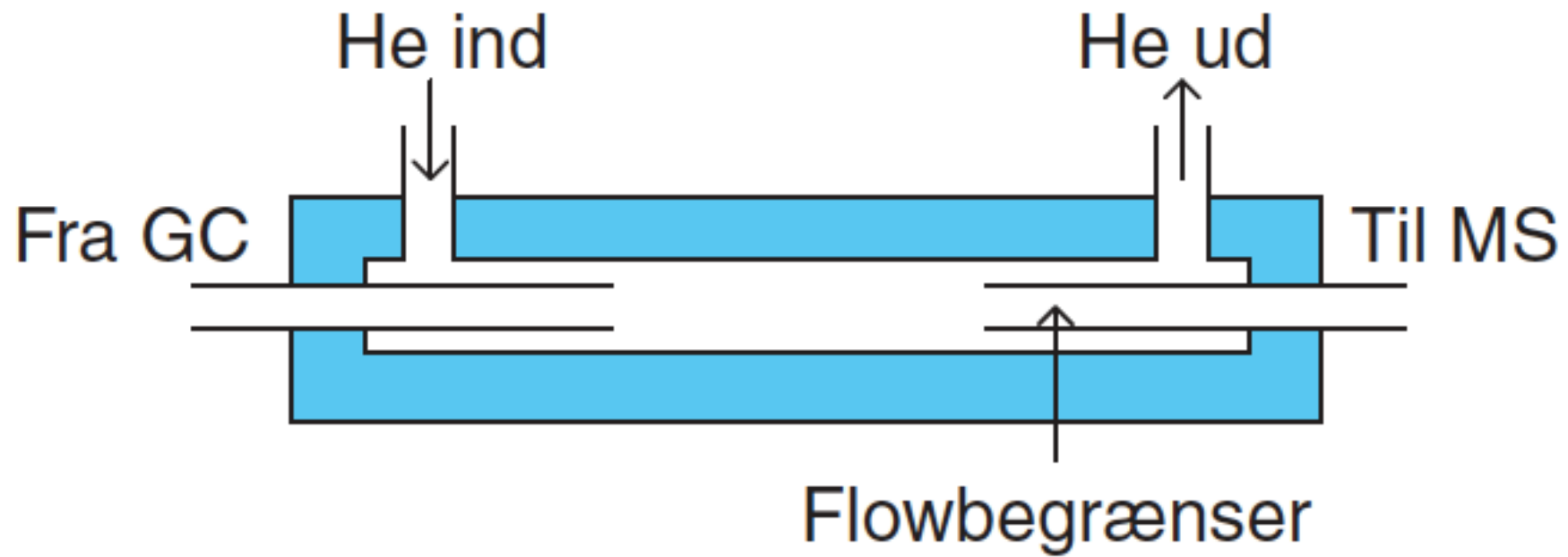


Figur 11.26. Eksempel på et ESI-TOF-spekter af CheY***.

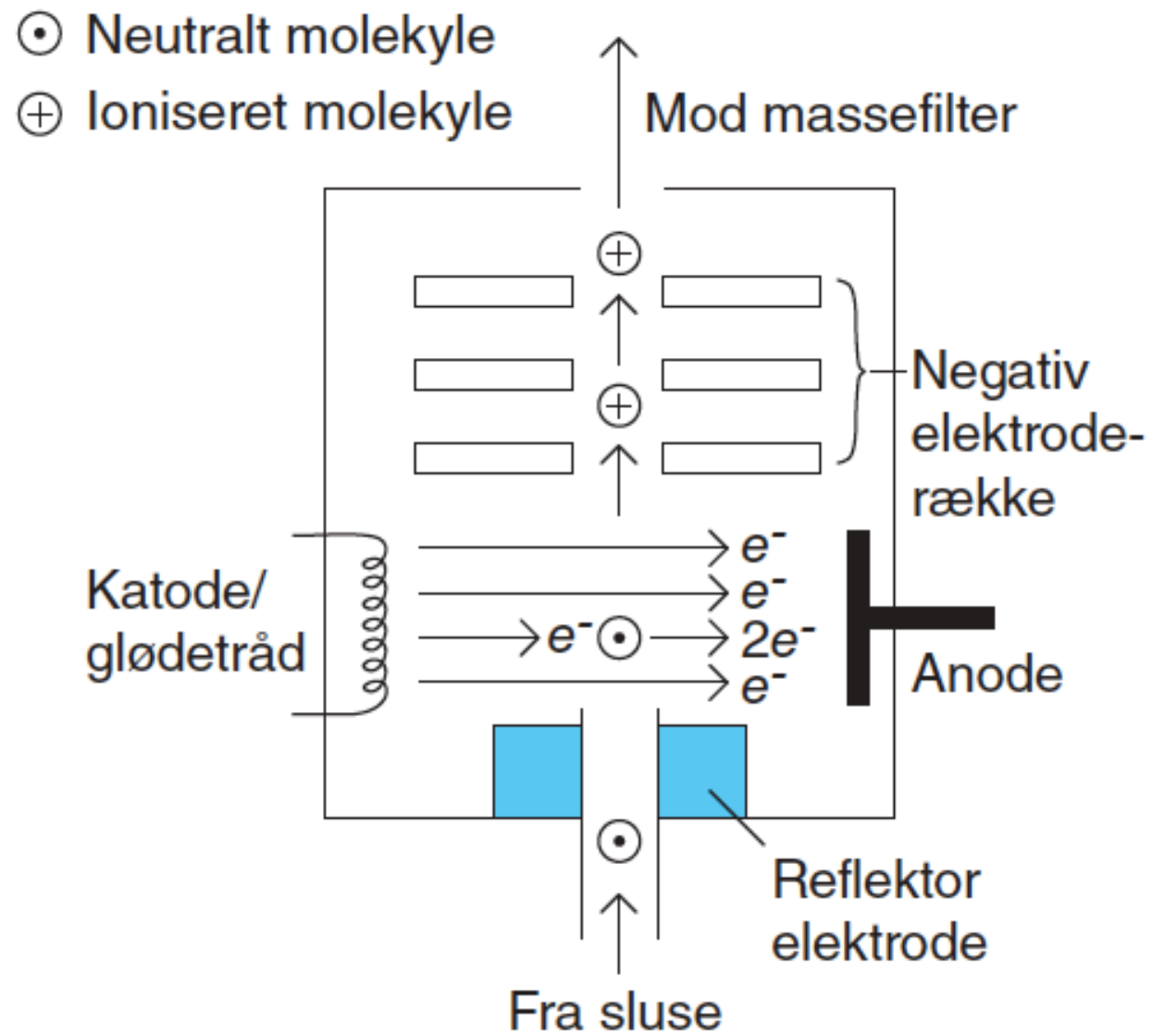
(Molarmassen af dette stof kan beregnes således: $z = 16$; $m/z = 941,81 \rightarrow m = 941,81 \cdot 16 = 15068,96$ Da)



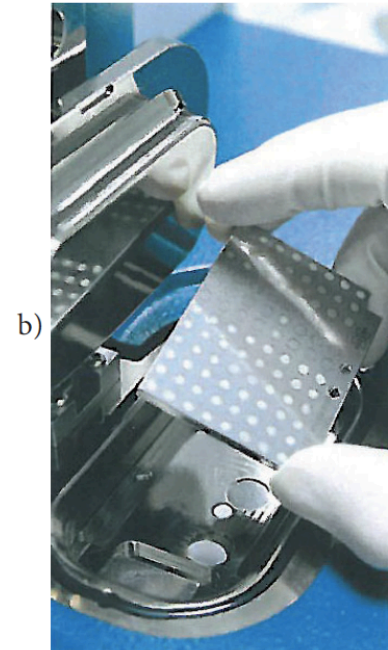
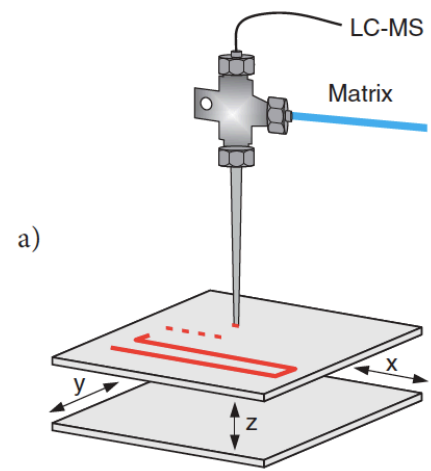
Figur 11.27. APCI-ionisering.



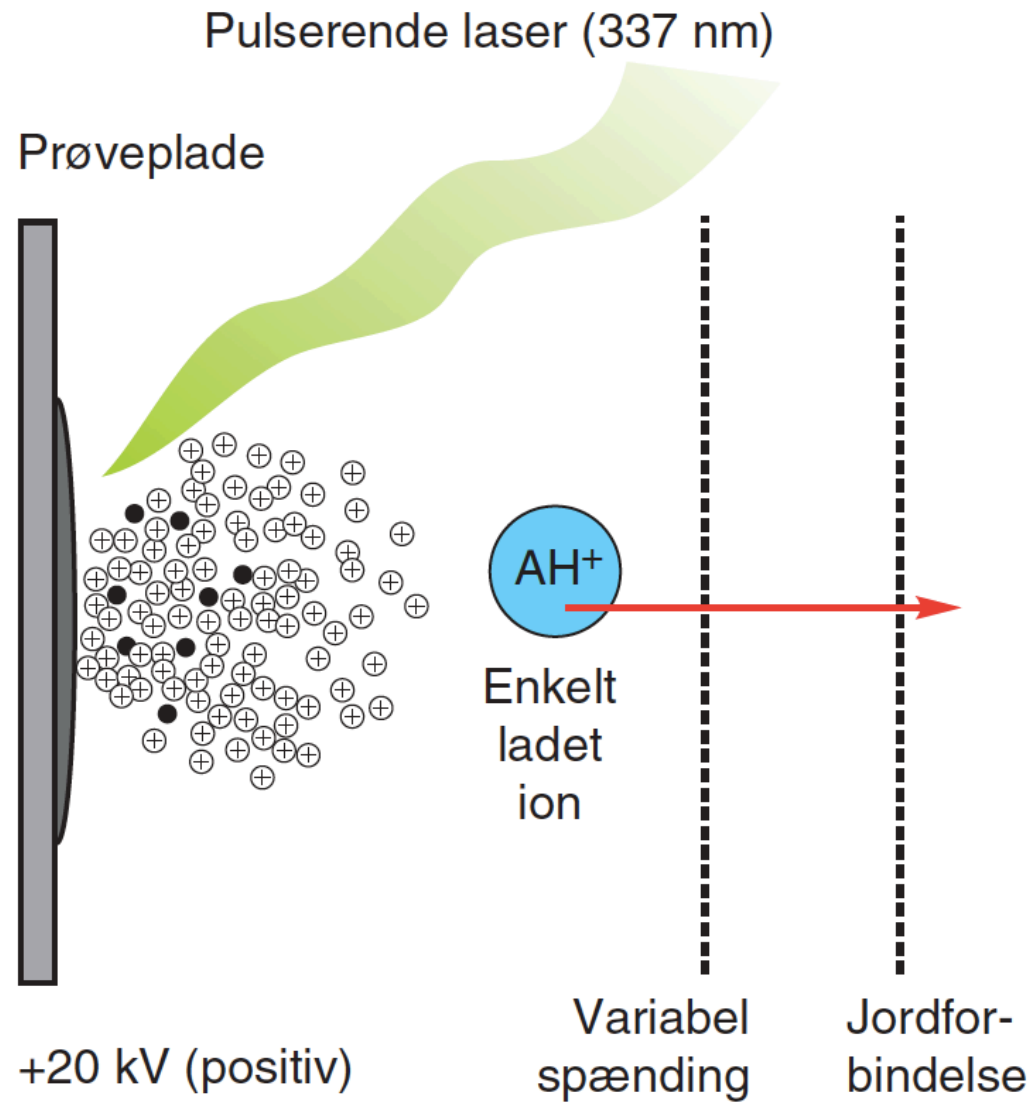
Figur 11.28. Split-interface til GC-MS.



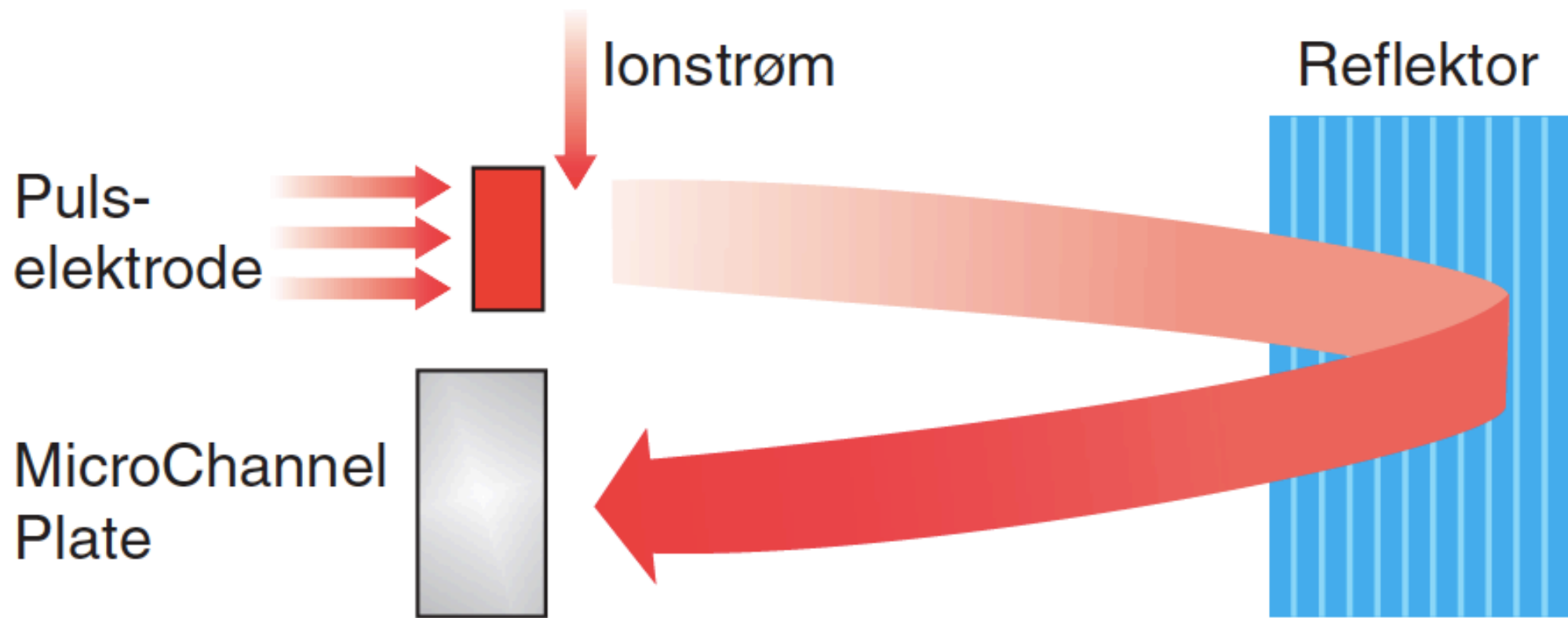
Figur 11.29. EI-ionkilde.



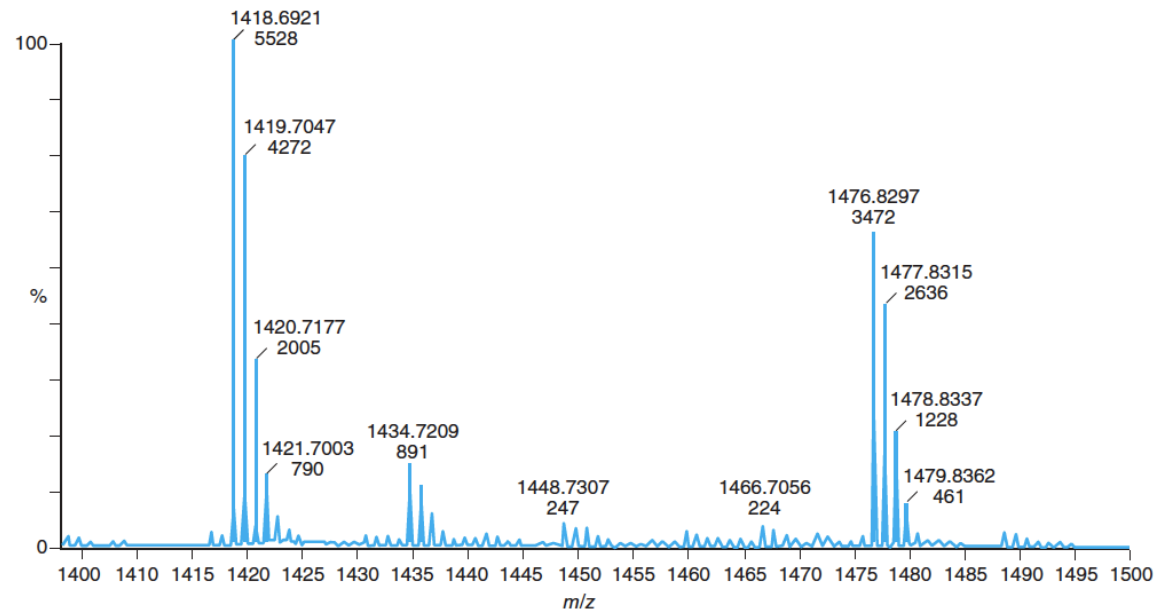
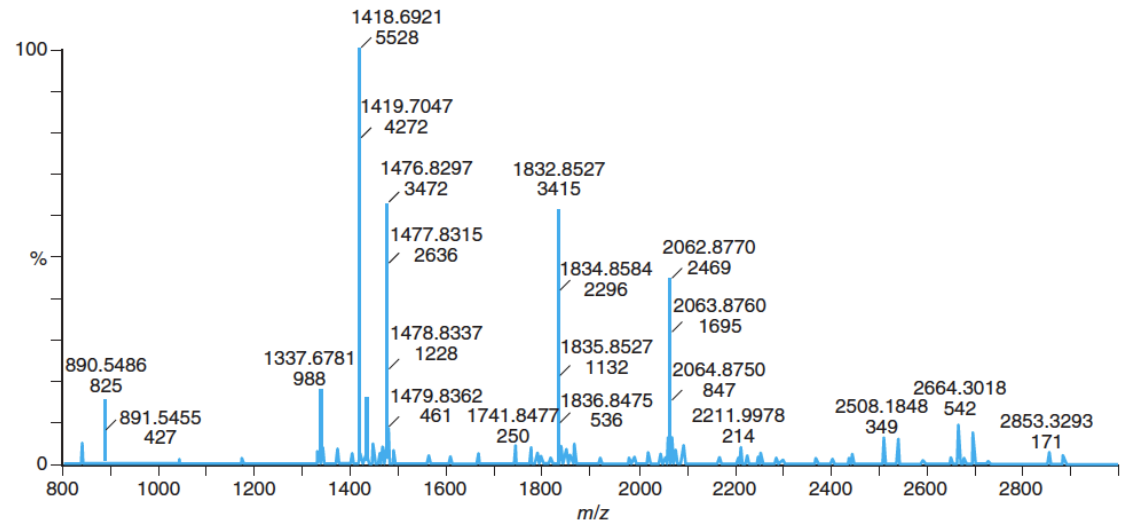
Figur 11.30. a) Automatisk påsætning af prøve.
b) Prøveintroduktion.



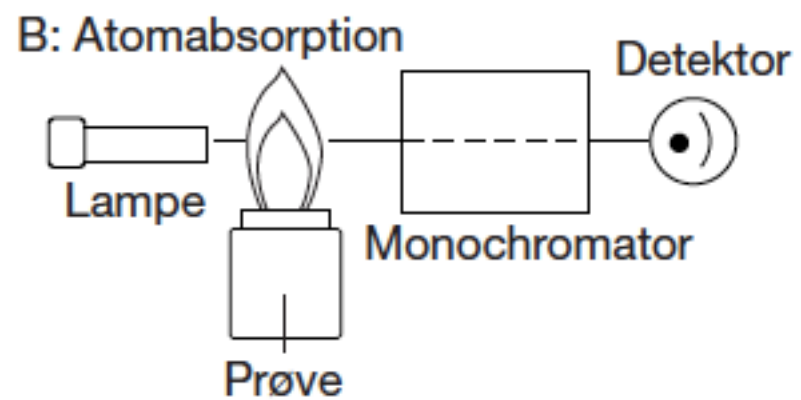
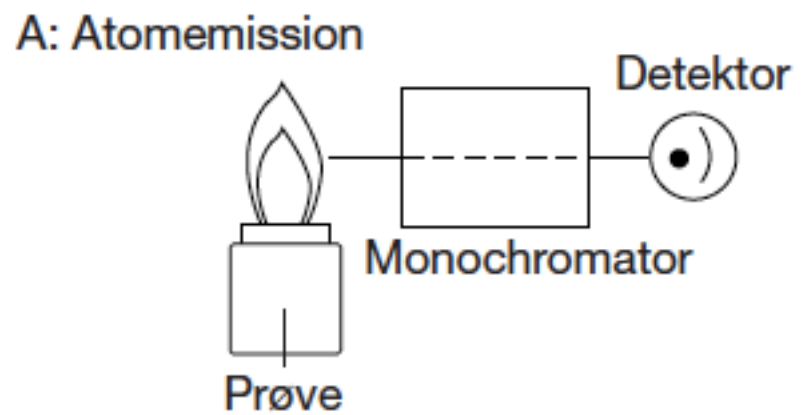
Figur 11.31. Ionisering med MALDI (positiv).



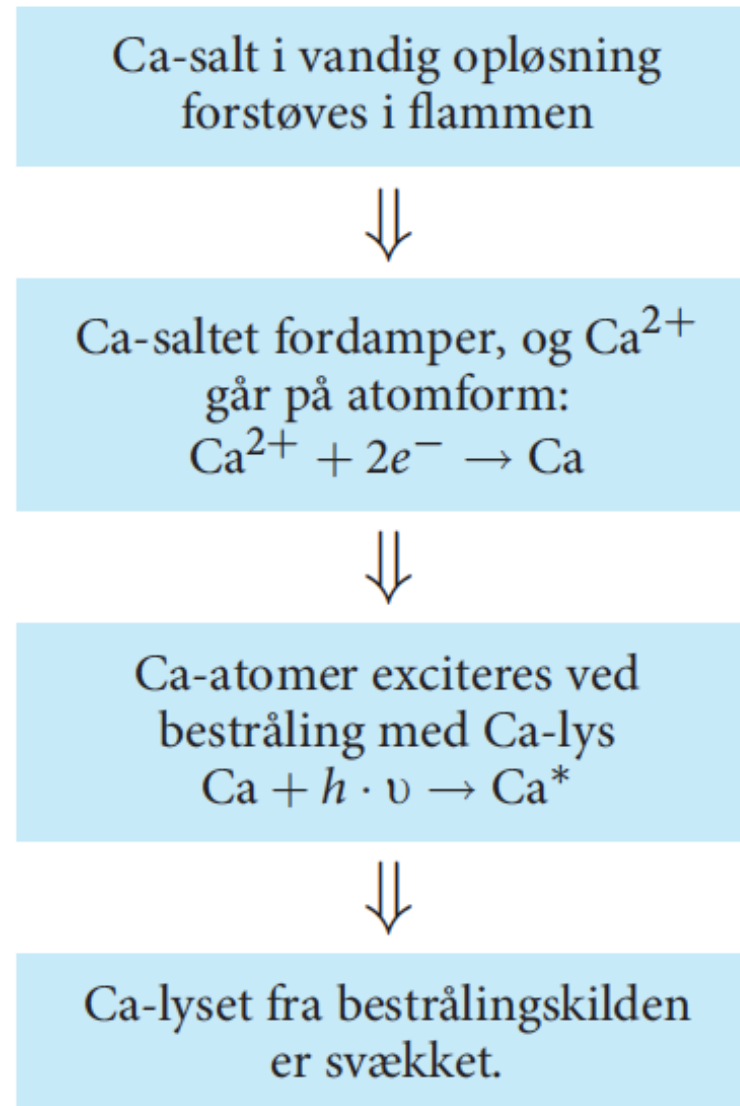
Figur 11.32. Skitse af rTOF.



Figur 11.33. a) Eksempel på et MALDI-TOF-spektrum scannet fra m/z 800-3000. b) Forstørrelse af området m/z 1400-1500, her ses en høj resolution (4 decimaler på m/z -værdien ved toppen af linjerne), der er en tydelig adskillelse mellem de enkelte isotoptoppe (et molekyle indeholder tre-fire ^{13}C -atomer). Tallet under m/z -værdien er TIC-værdien.



Figur 12.1. Forskellen på emission og absorption.



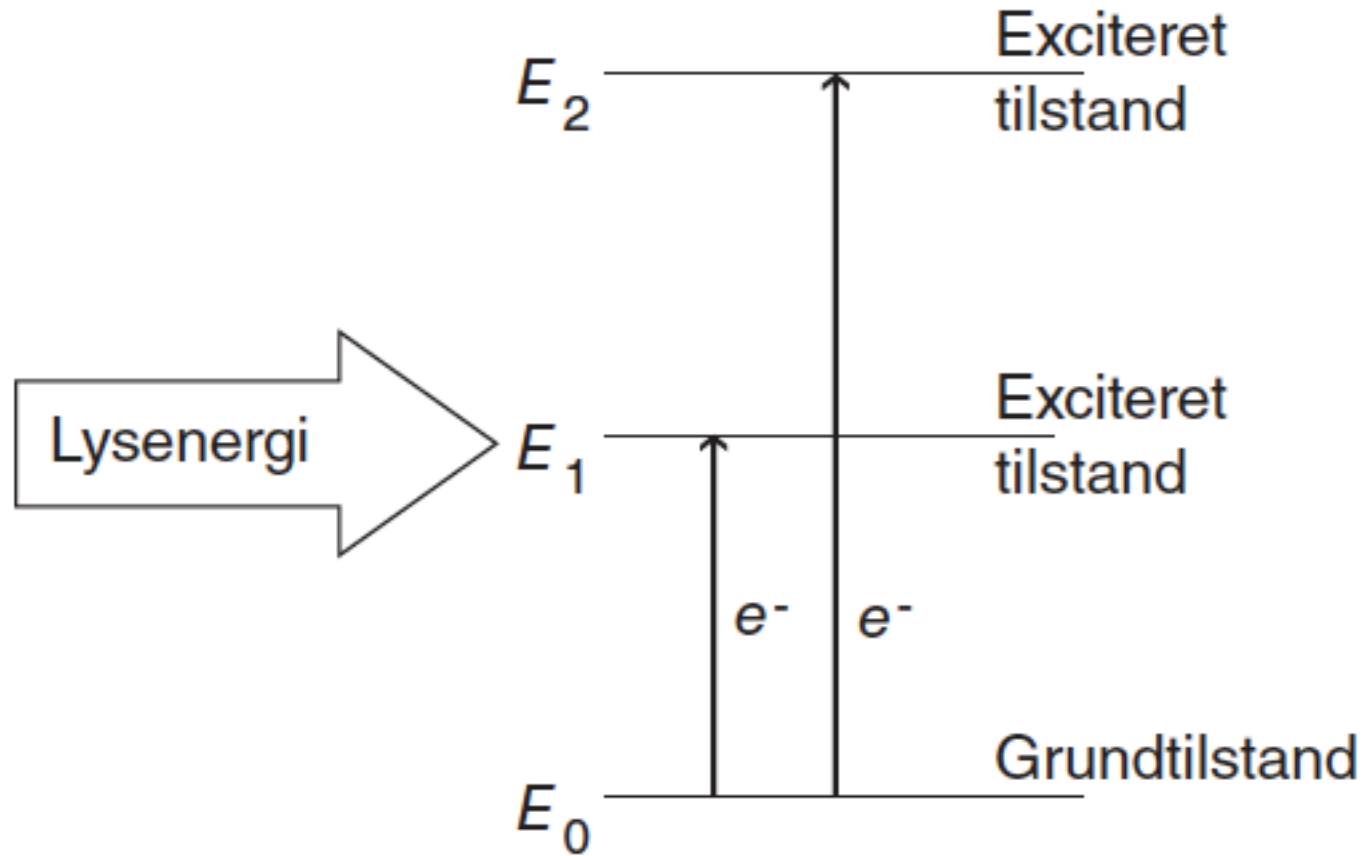
Figur 12.2. Processerne i flammen.

<i>Metal</i>	<i>% p atomform⁹⁵ ved 2400 °C</i>
Lithium	99,99 %
Natrium	98,9 %
Kalium	90,7 %
Calcium	99,99 %

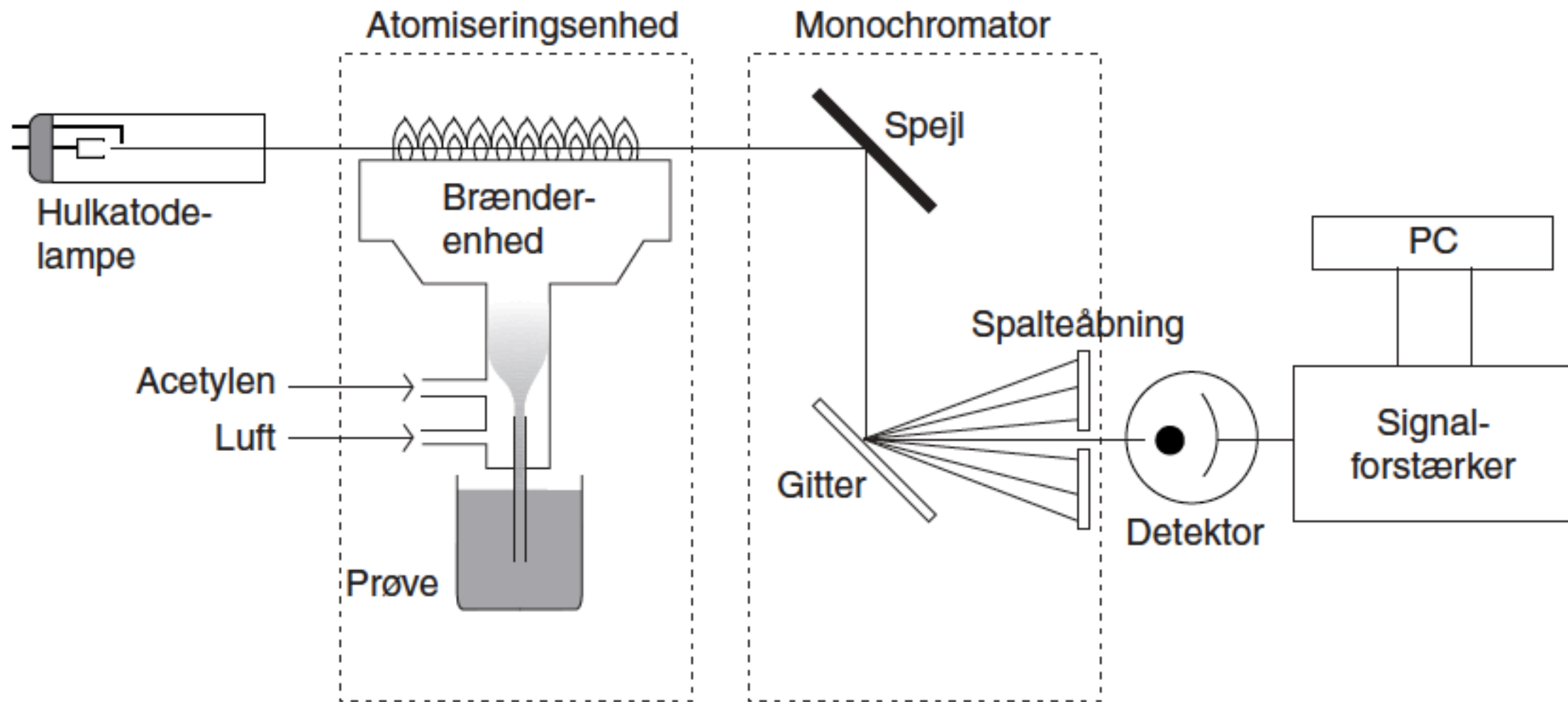
Tabel 12.1.

<i>Metal</i>	<i>1727 °C</i>	<i>2727 °C</i>
Ca*:Ca	1:10 ⁷	1:10 ⁴
Cu*:Cu	1:10 ¹⁰	1:10 ⁸
Zn*:Zn	1:10 ¹⁴	1:10 ⁷

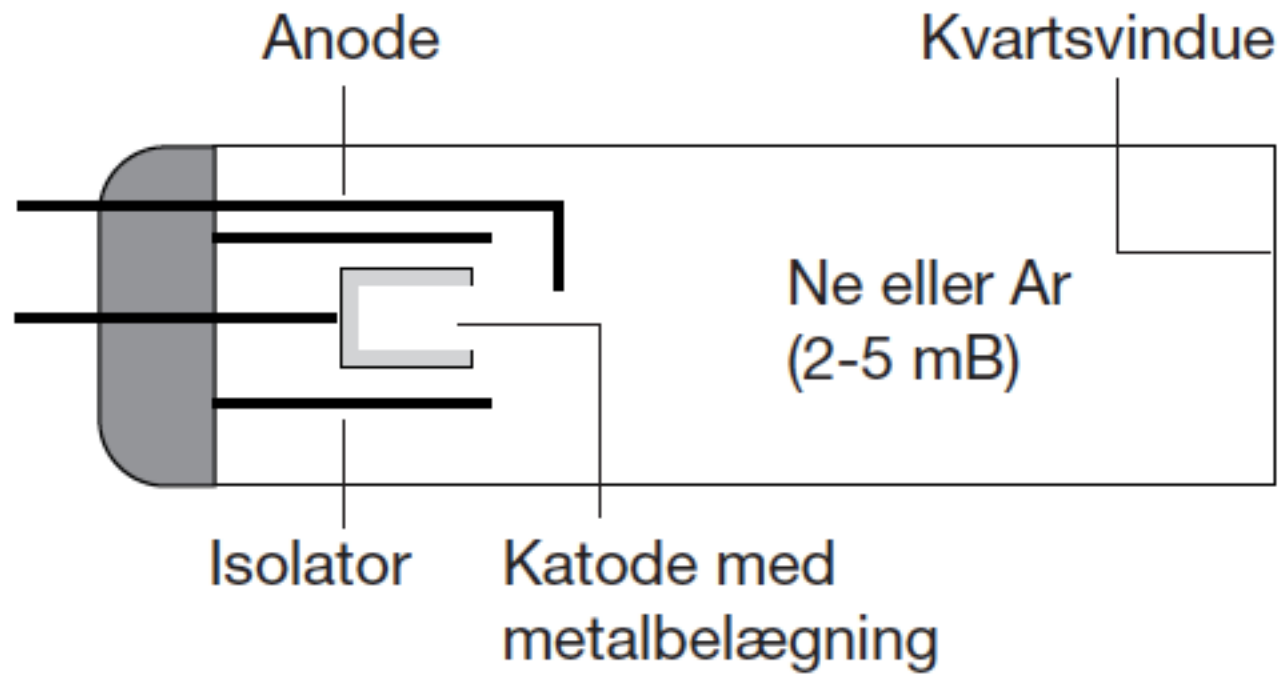
Tabel 12.2.



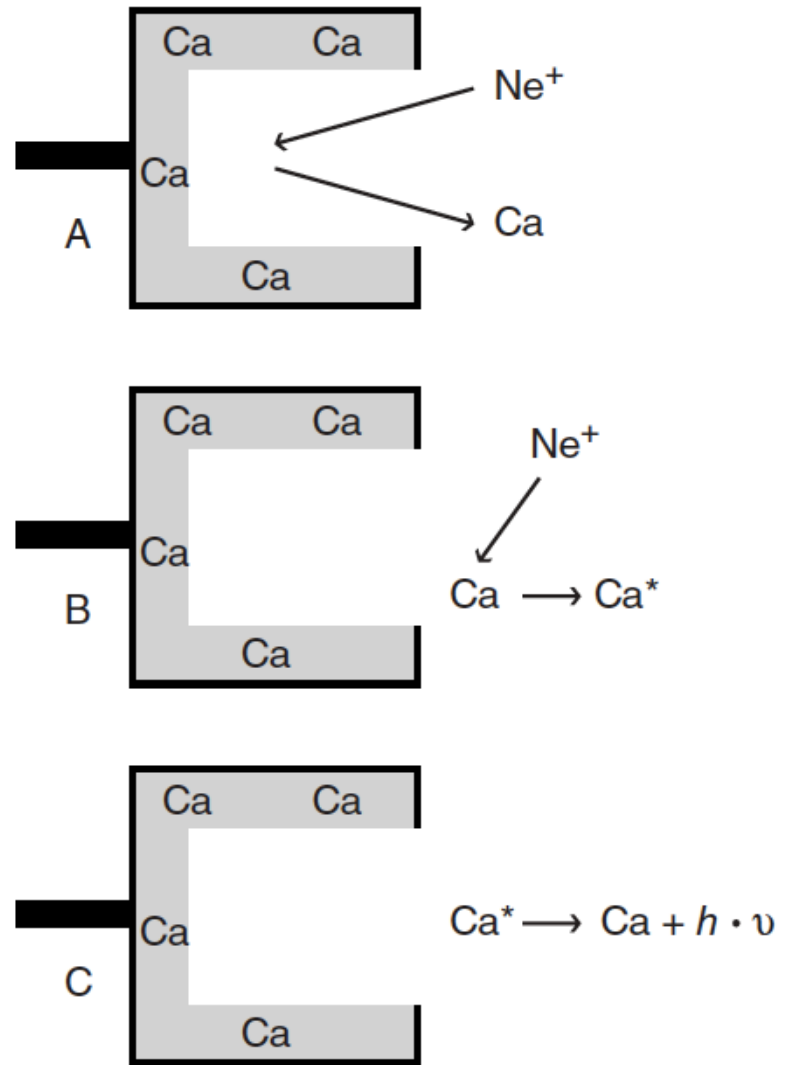
Figur 12.3. Excitering af elektronerne i et atom.



Figur 12.4. Atomabsorptionsspektrofotometrets opbygning.



Figur 12.5. Hulkatodelampe.



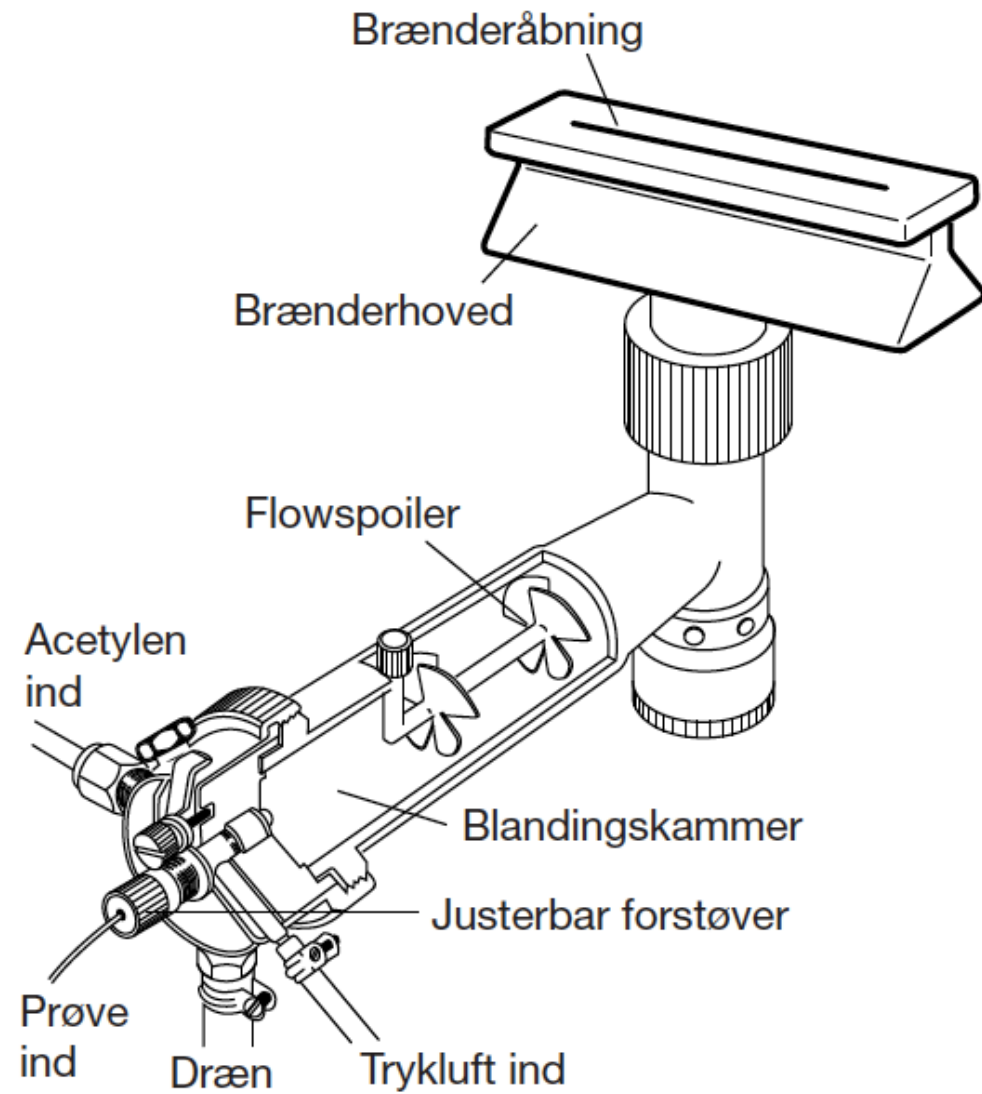
Figur 12.6. Lysudsendelse fra hulkatoden.

<i>Element</i>	<i>Linjespektrum (nm)</i>
Calcium	422,7 og 239,9
Bly	202,2 - 205,3 - 217,0 - 261,4 - 283,3 - 364,0 - 368,3

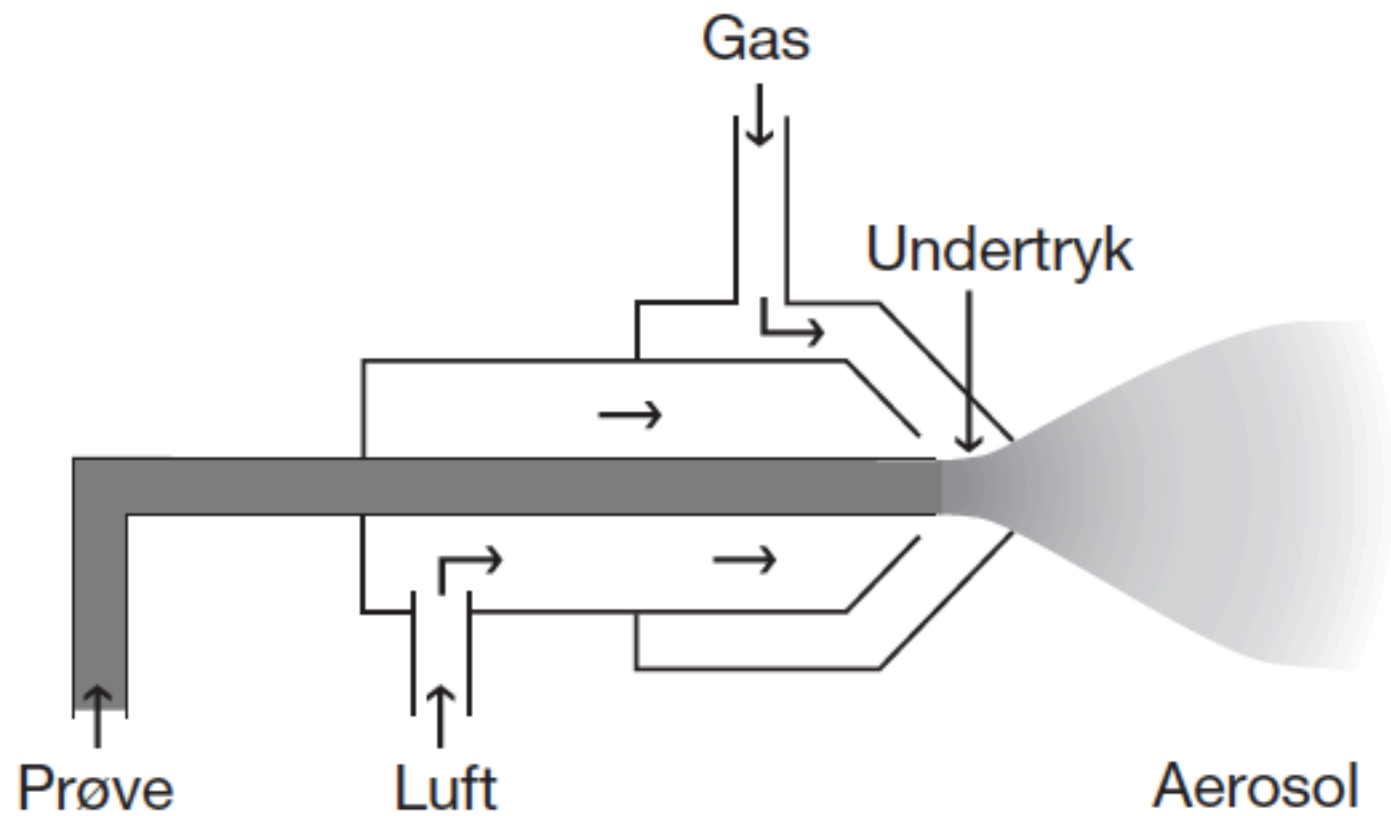
Tabel 12.3.

<i>Element</i>	λ (nm)	<i>Detektionsgrænse (ppb)</i>
Ca	422,7	2
Cr	357,9	3
Fe	248,3	4
Mg	285,2	0,3
Ni	232,0	6
Pb	217,0	8
Sn	224,6	24
Zn	213,9	0,9

Tabel 12.4.



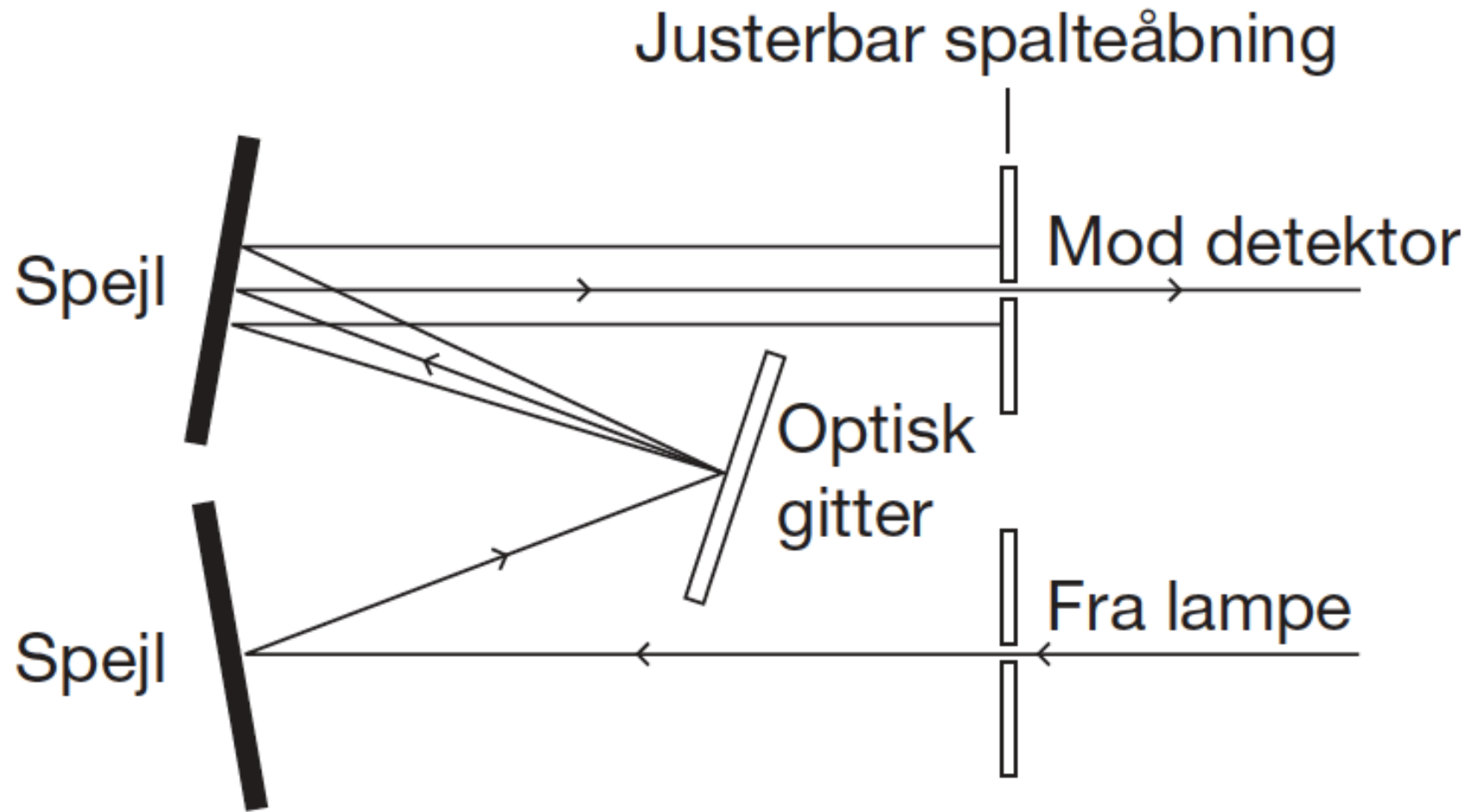
Figur 12.7. Atomiseringsenheden med forstøver og brænder.



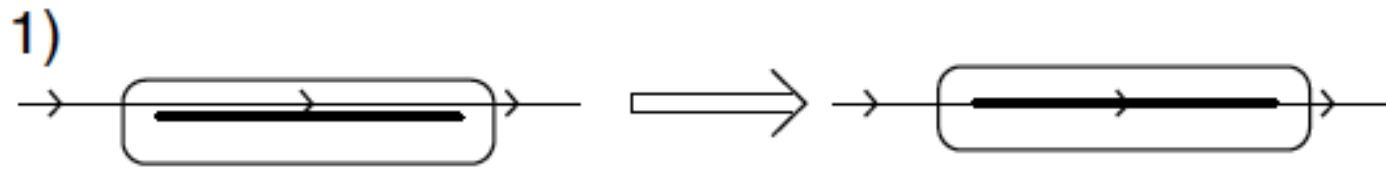
Figur 12.8. Forstøverenheten.

<i>Brændstof</i>	<i>Oxidant</i>	<i>Flammen °C</i>
Propan	Atm. luft	1900
Acetylen	Atm. luft	2400
Acetylen	Lattergas	2800
Acetylen	Oxygen	3200

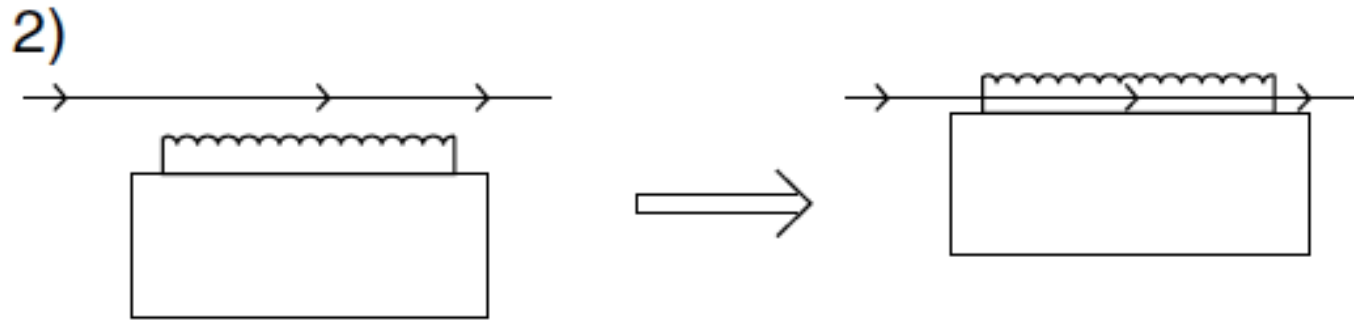
Tabel 12.5.



Figur 12.9. Monochromatorens opbygning.



Brænderhoved set ovenfra, det flyttes vandret indad i forhold til lysstrålen



Brænderhoved set fra siden, det hæves i forhold til lysstrålen

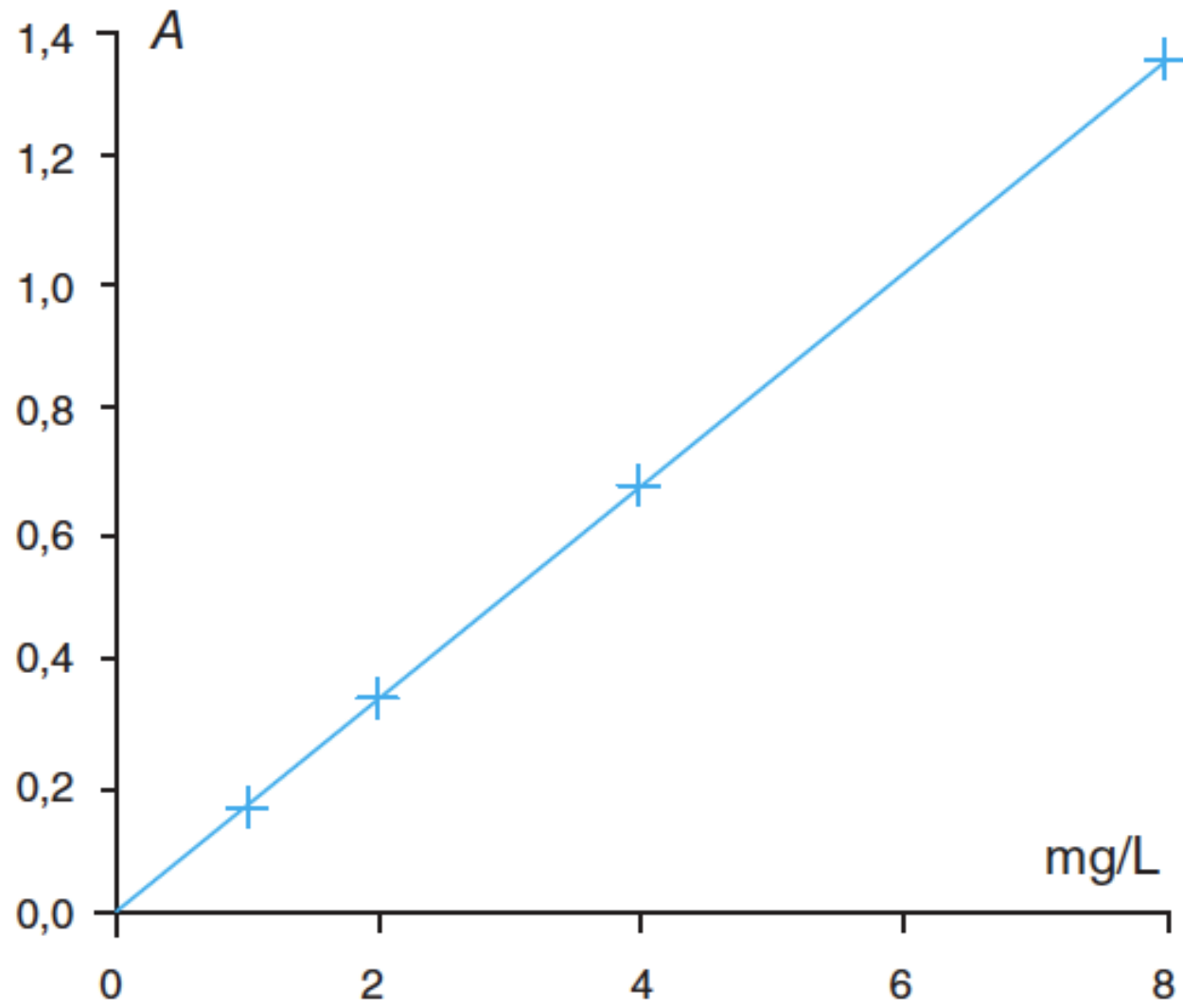


Brænderhoved set ovenfra, det drejes i forhold til lysstrålen

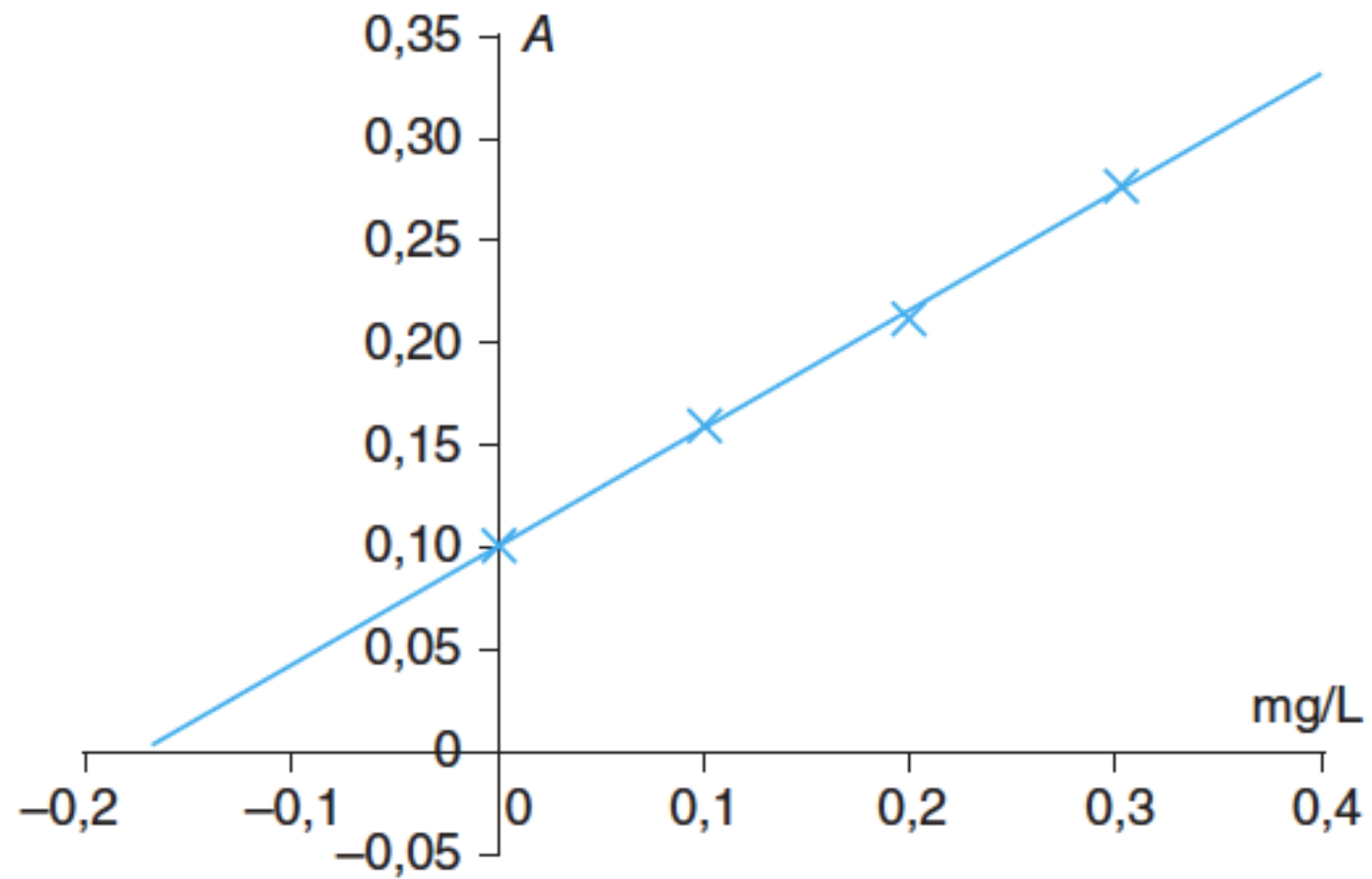
Figur 12.10. Justering af brænderhovedet.

<i>Element</i>	<i>Sensitivity (mg/L)</i>
Bly	0,055
Calcium	0,077
Cadmium	0,026
Chrom	0,088
Jern	0,132
Kviksølv	7,70
Magnesium	0,007
Tin	3,96
Zink	0,018

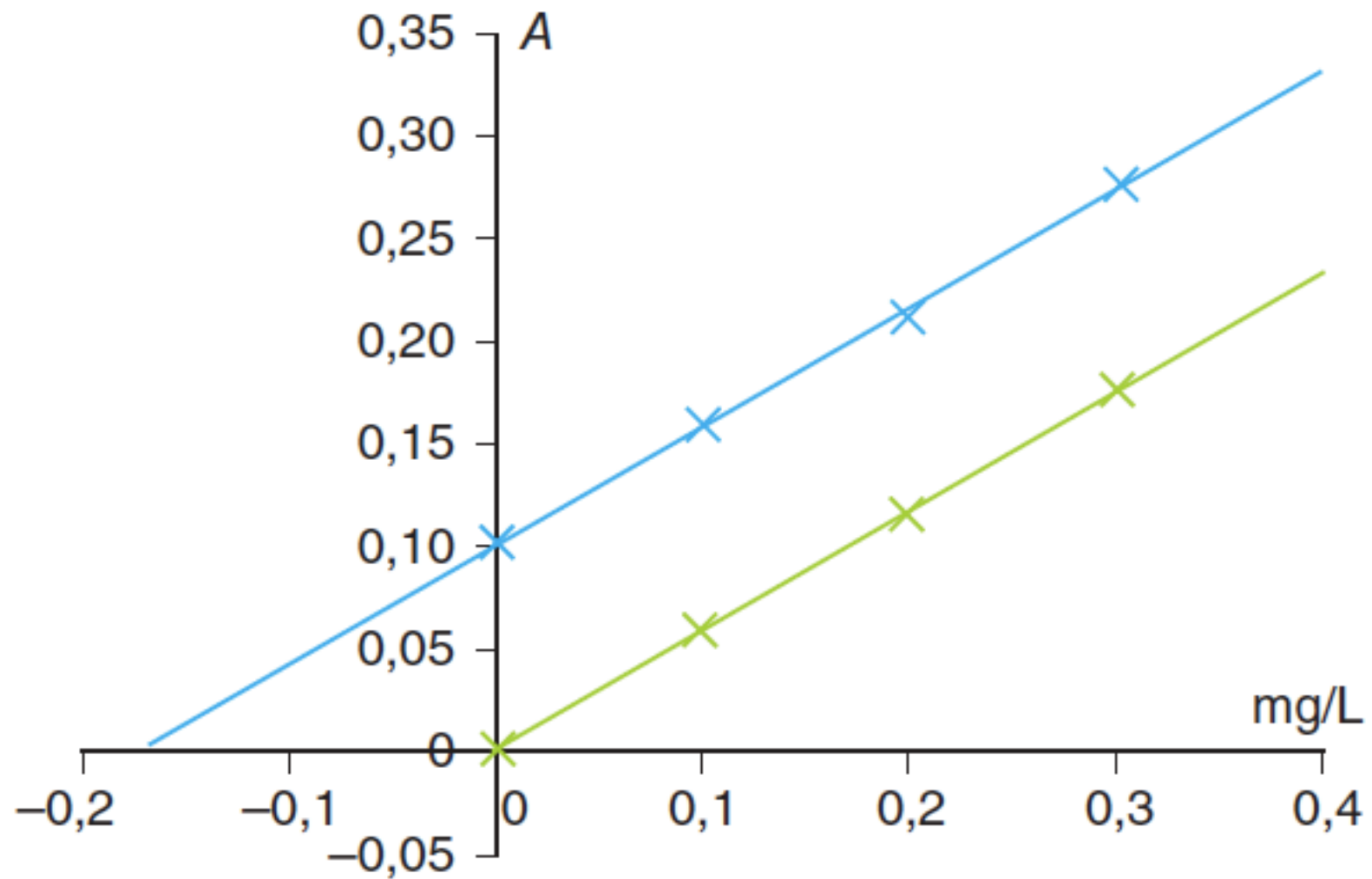
Tabel 12.6.



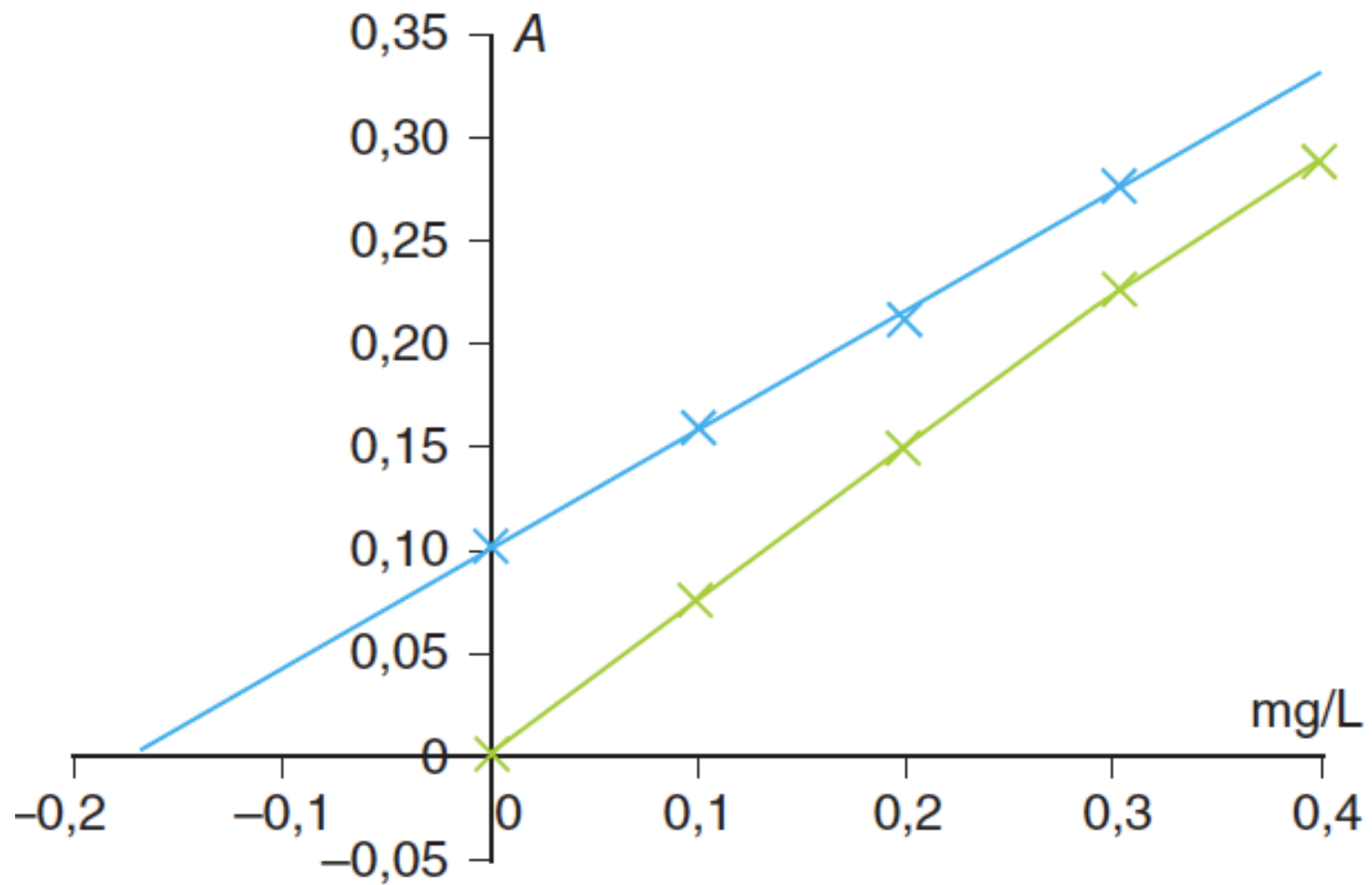
Figur 12.11. Standardkurve, AAS.



Figur 12.12. Standardadditionskurve, AAS.



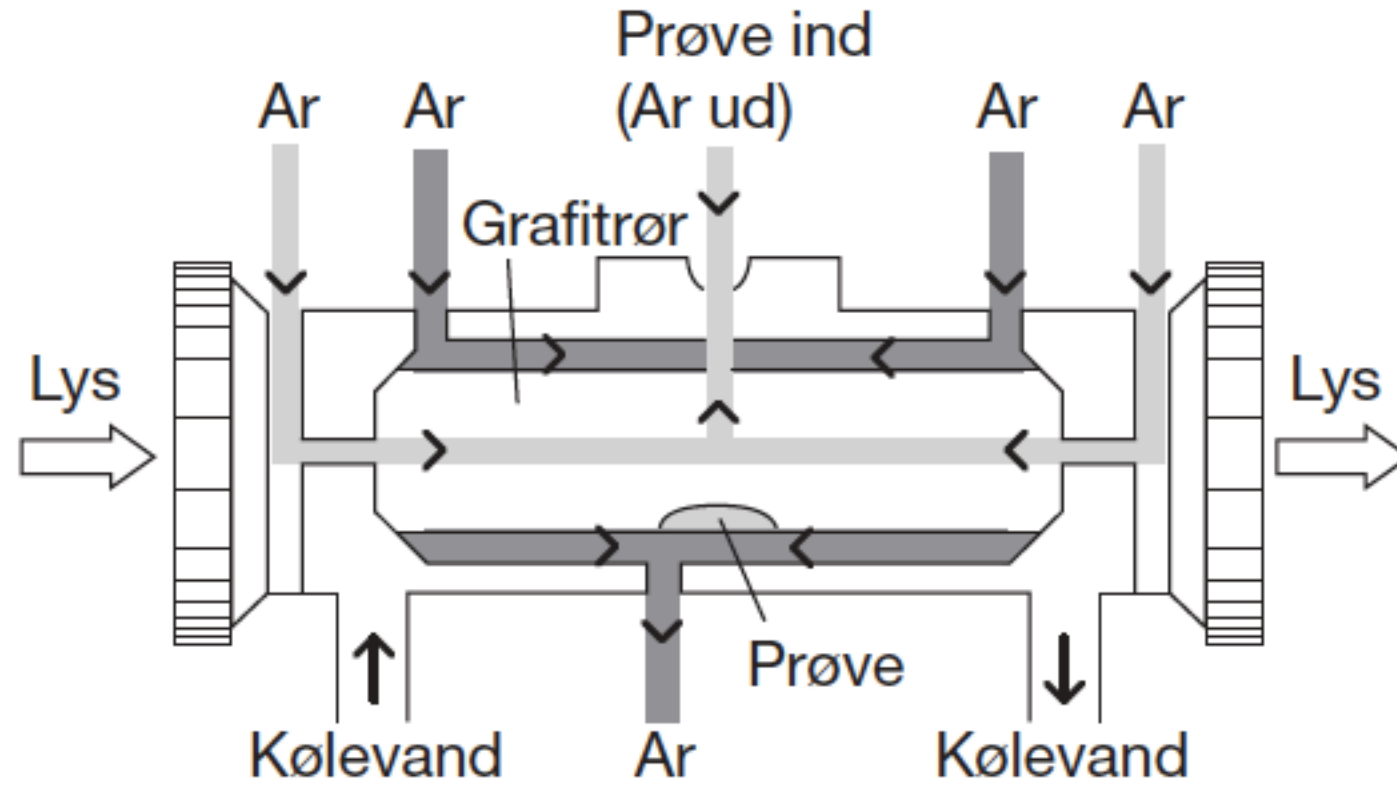
Figur 12.13. Additionskurve uden interferens.



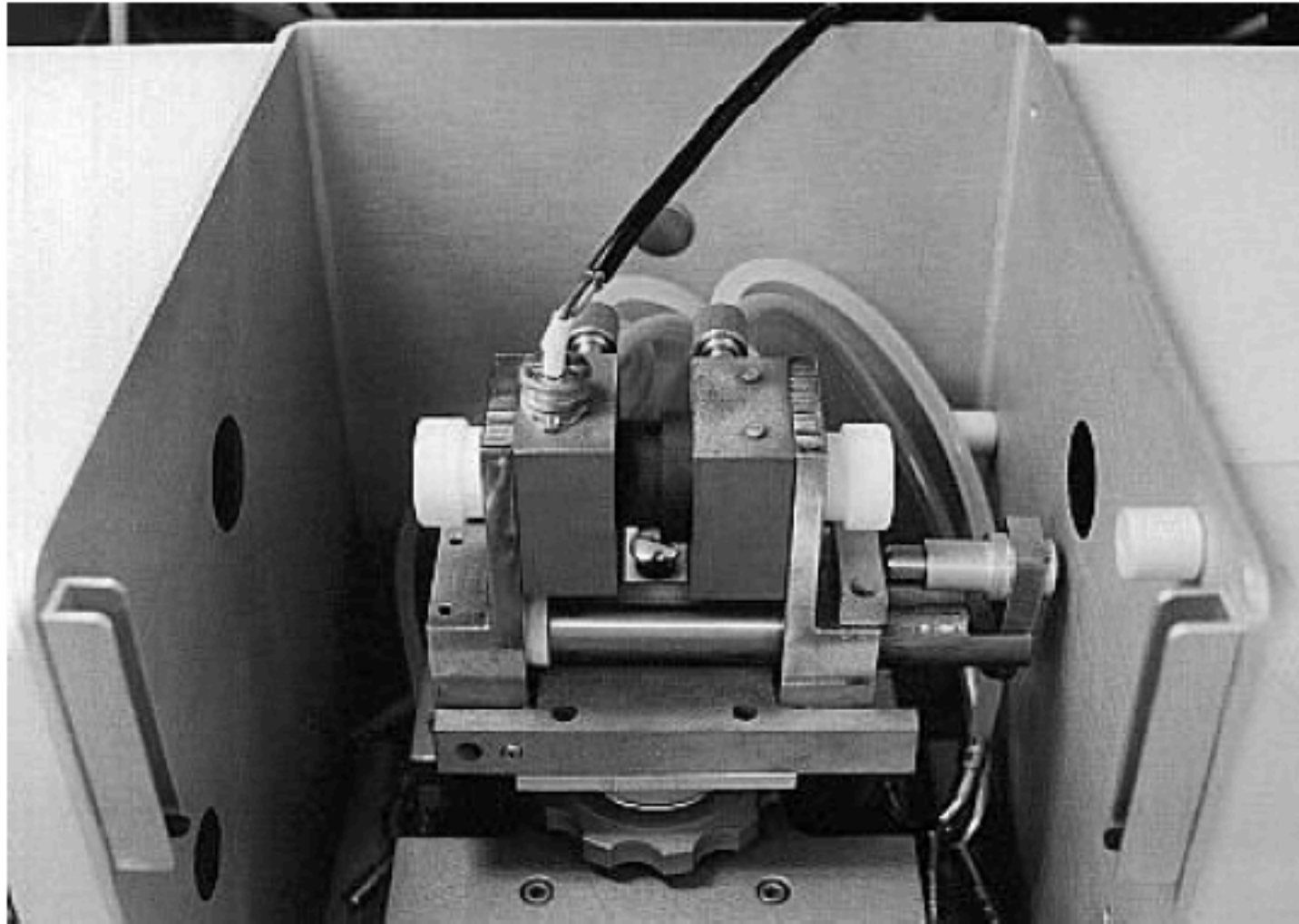
Figur 12.14. Additionskurve med interferens.

<i>Element</i>	<i>acetylen/ luft</i>	<i>acetylen/ lattergas</i>	<i>acetylen/ oxygen</i>
Natrium	1 %	-	26 %
Kalium	9 %	-	82 %
Magnesium	-	6 %	0,01 %
Calcium	0,01 %	43 %	7 %
Barium	2 %	88 %	42 %

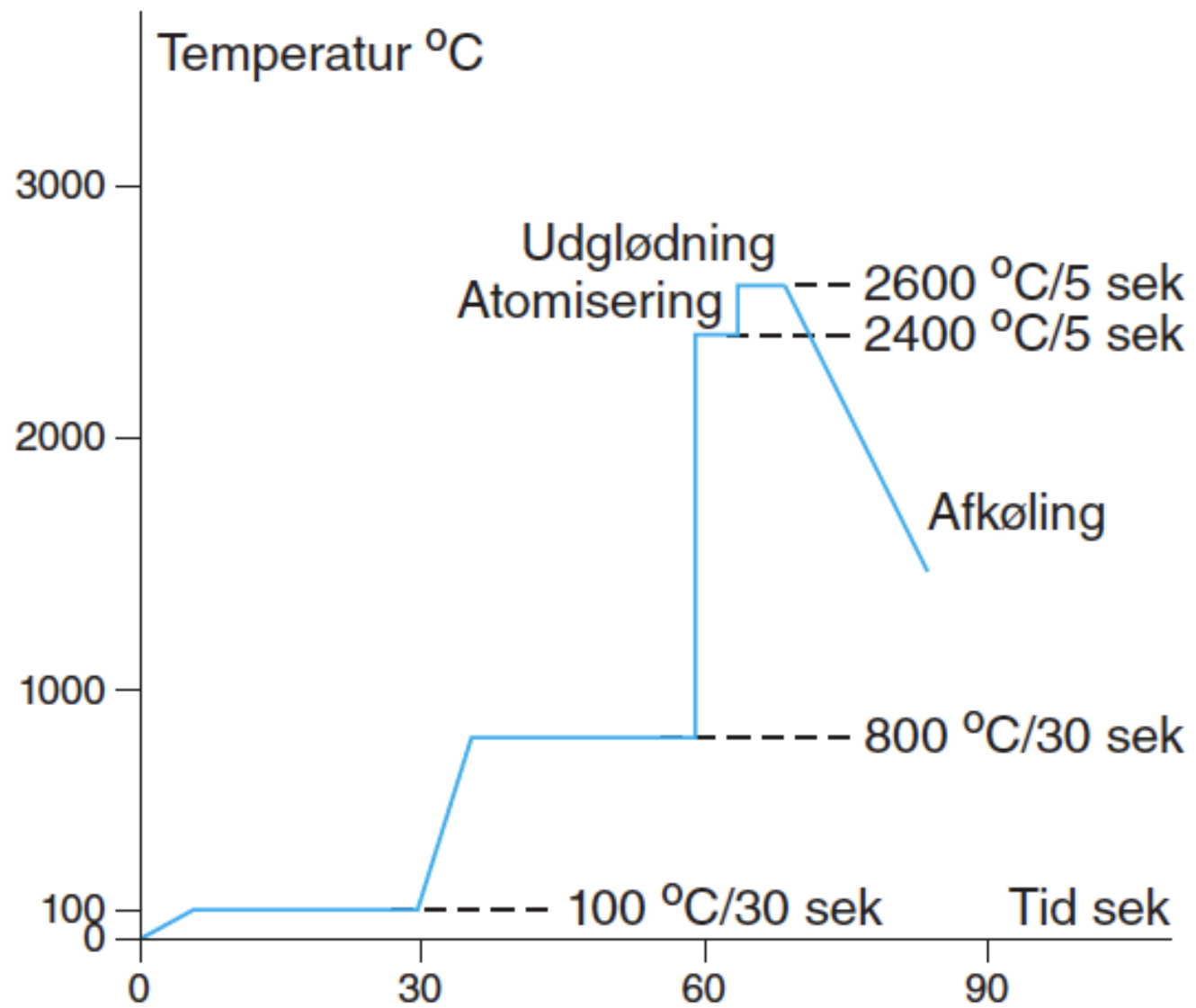
Tabel 12.7. Procent af atomerne, der er på ionform.



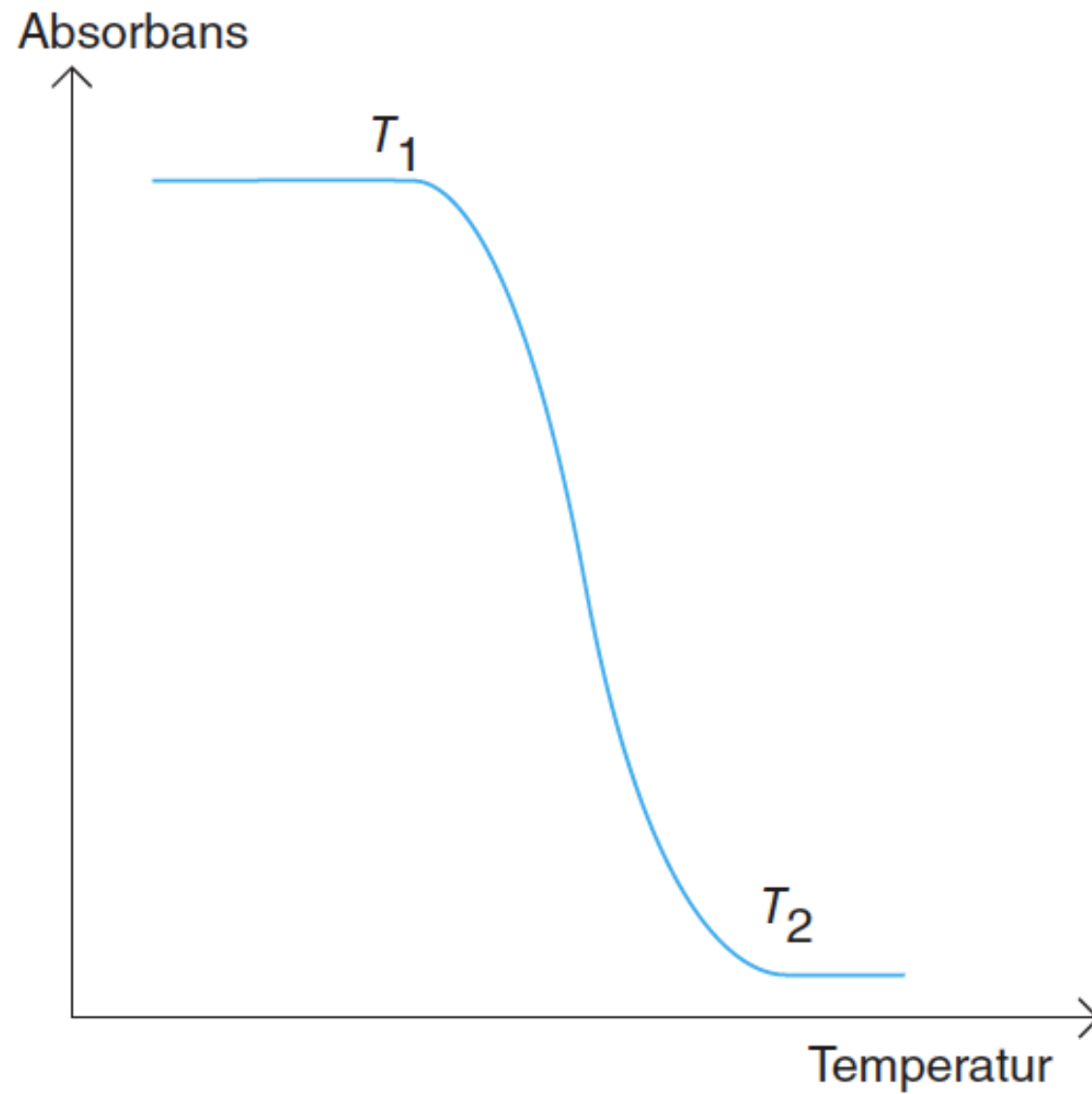
Figur 12.15. Grafitrør placeret i holderen.



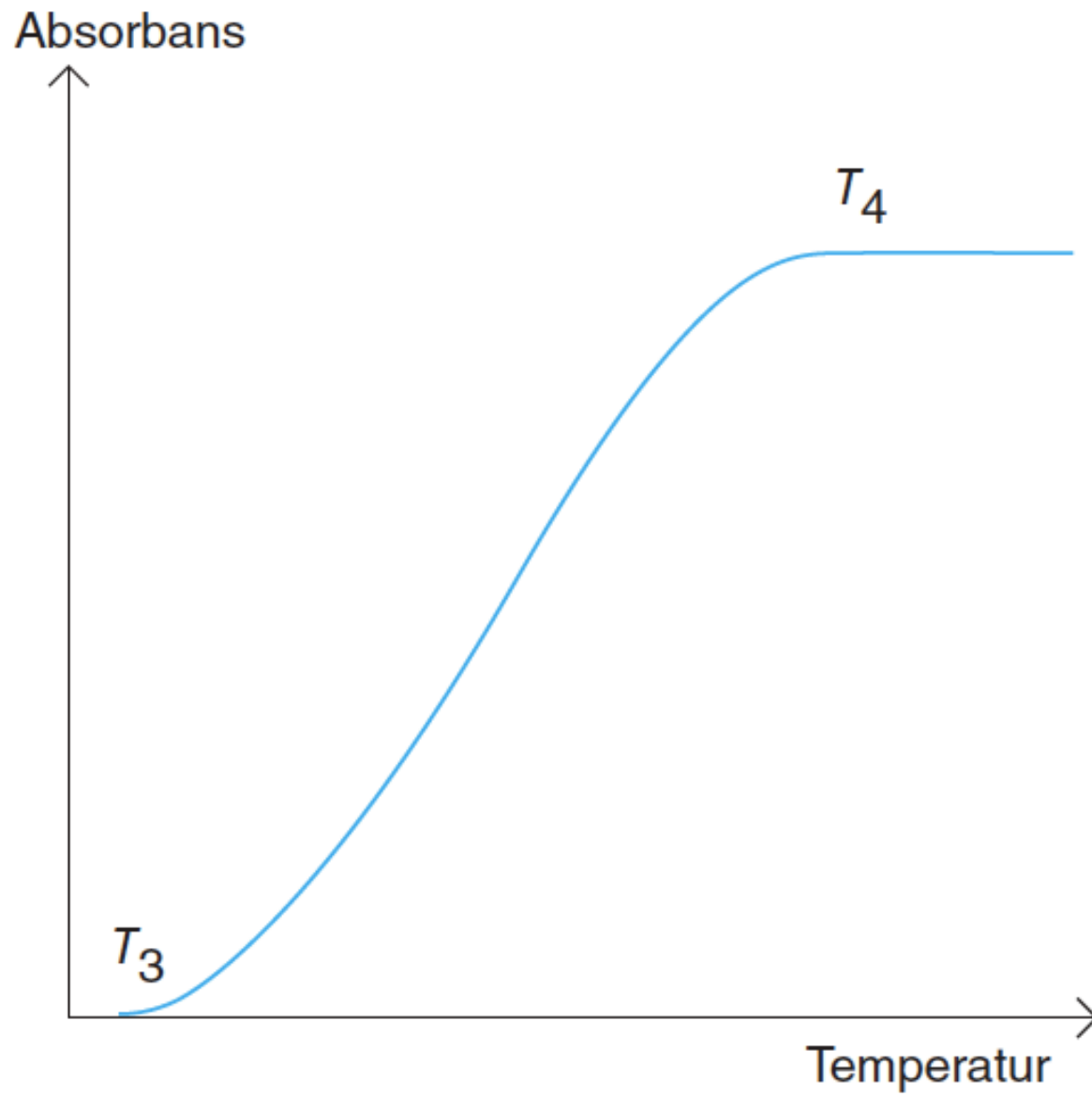
Figur 12.16. Brænderen erstattet af grafitovn.



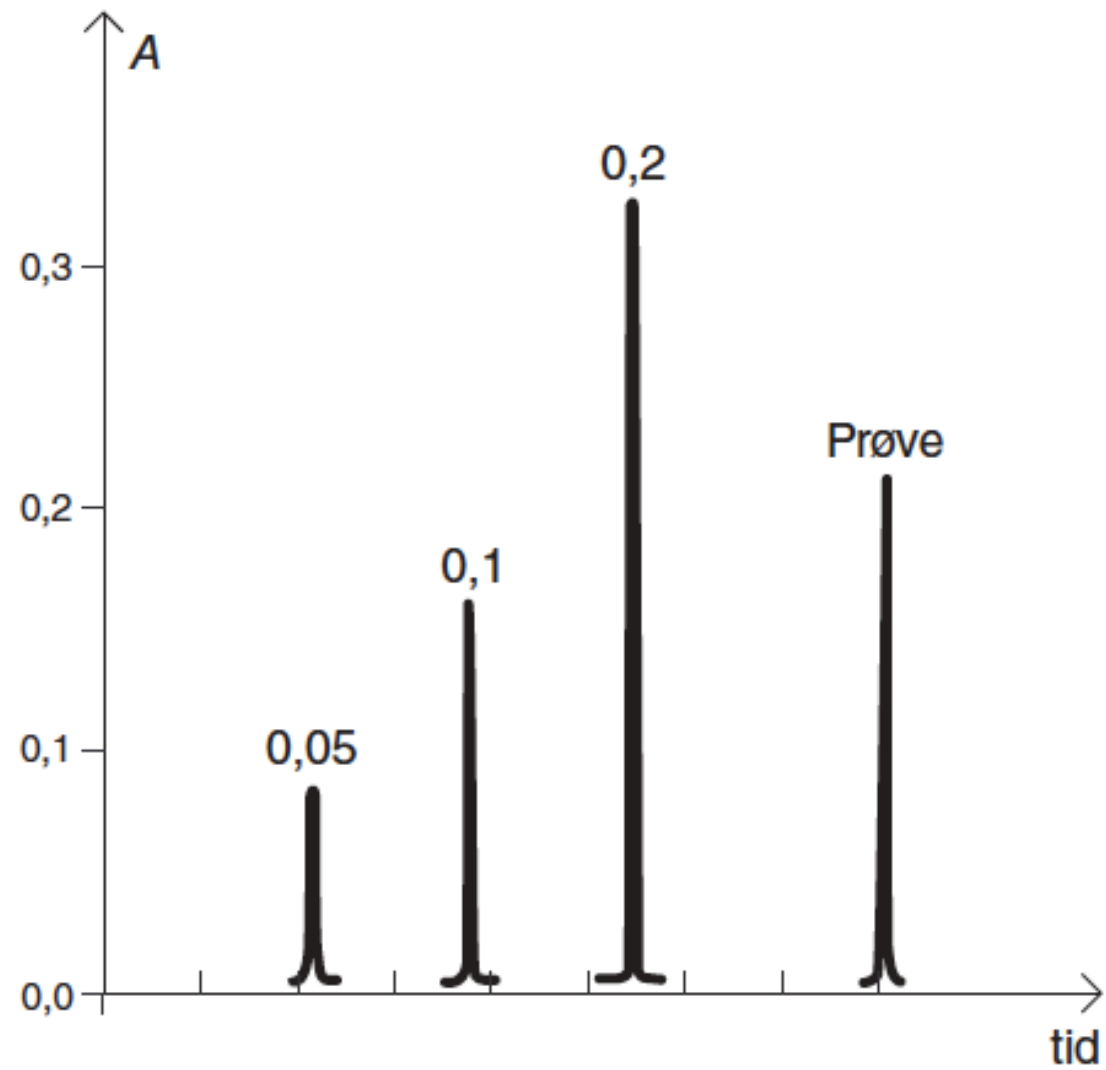
Figur 12.17. Grafitovnsprogram.



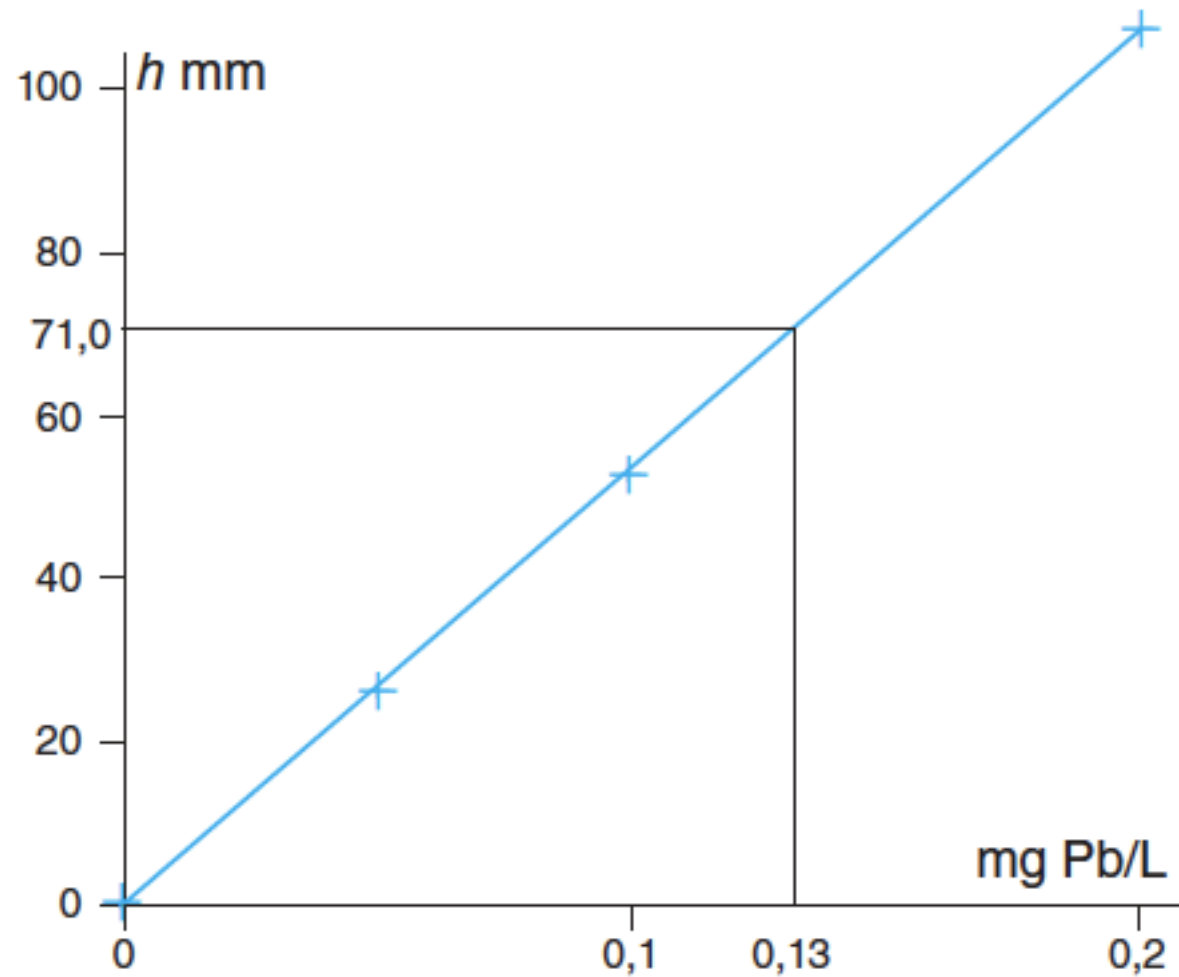
Figur 12.18. Optimering af forskningstemperaturen.



Figur 12.19. Optimering af atomiseringstemperaturen.



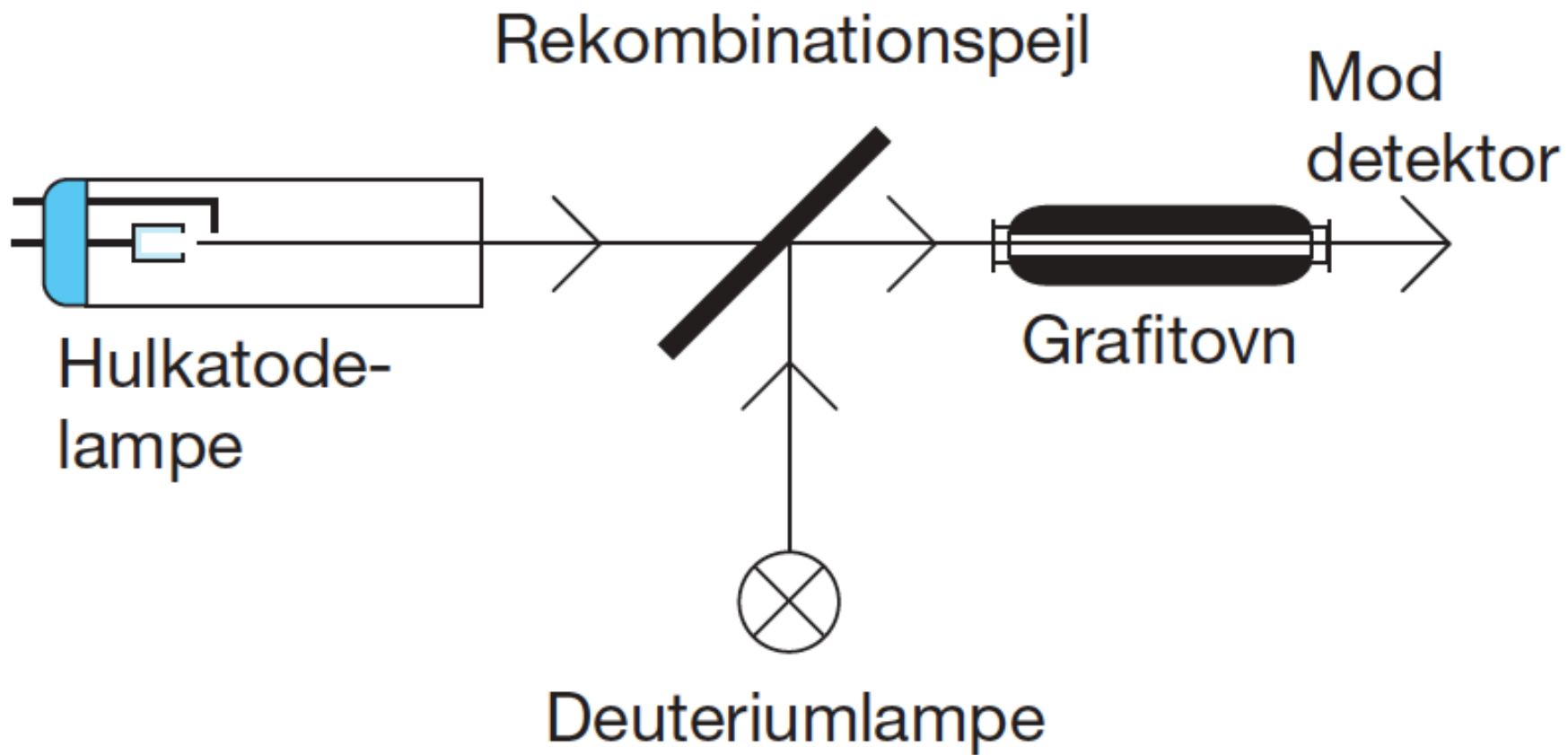
Figur 12.20. Optegning af resultater fra måling med grafitovn.



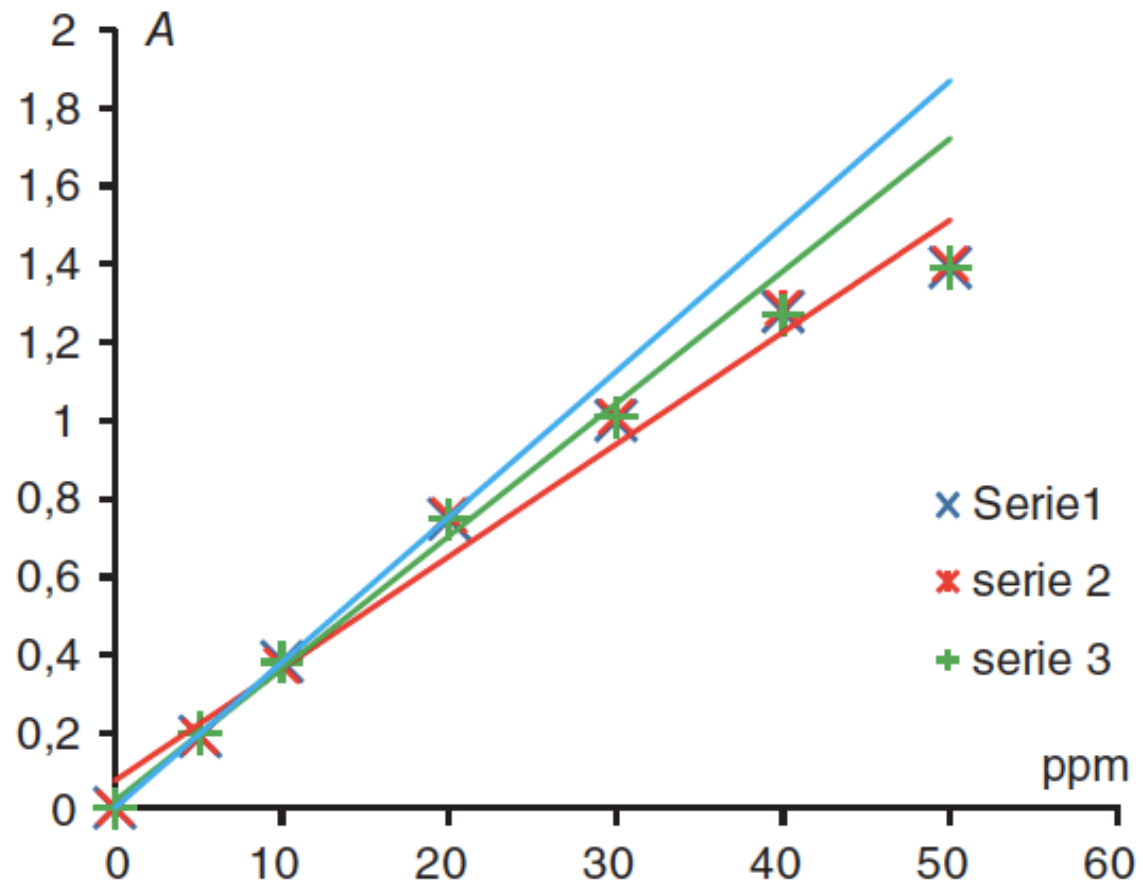
Figur 12.21. Standardkurve fra måling med grafitovn.

<i>Element</i>	λ (nm)	<i>Flamme (ppm)</i>	<i>Grafitovn (ppm)</i>
Ca	422,7	2	0,05
Cr	357,9	3	0,01
Fe	248,3	4	0,02
Mg	285,2	0,3	0,004
Ni	232,0	6	0,2
Pb	217,0	8	0,05
Sn	224,6	24	0,1
Zn	213,9	0,9	0,001

Tabel 12.8.



Figur 12.22. Baggrundskorrektorens placering i AAS-strålegangen.

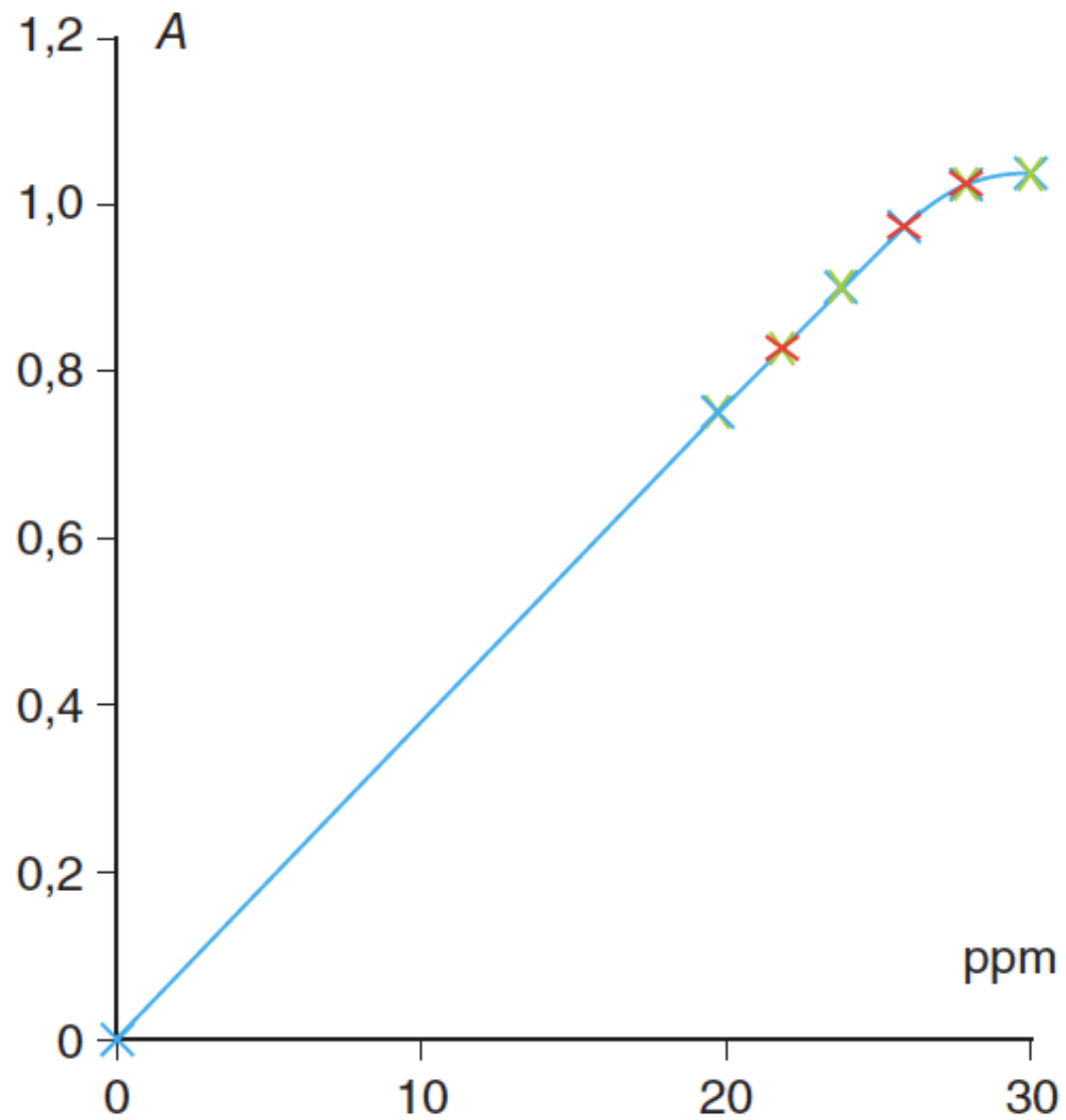


Figur 12.23. Visuel vurdering af linearitet.

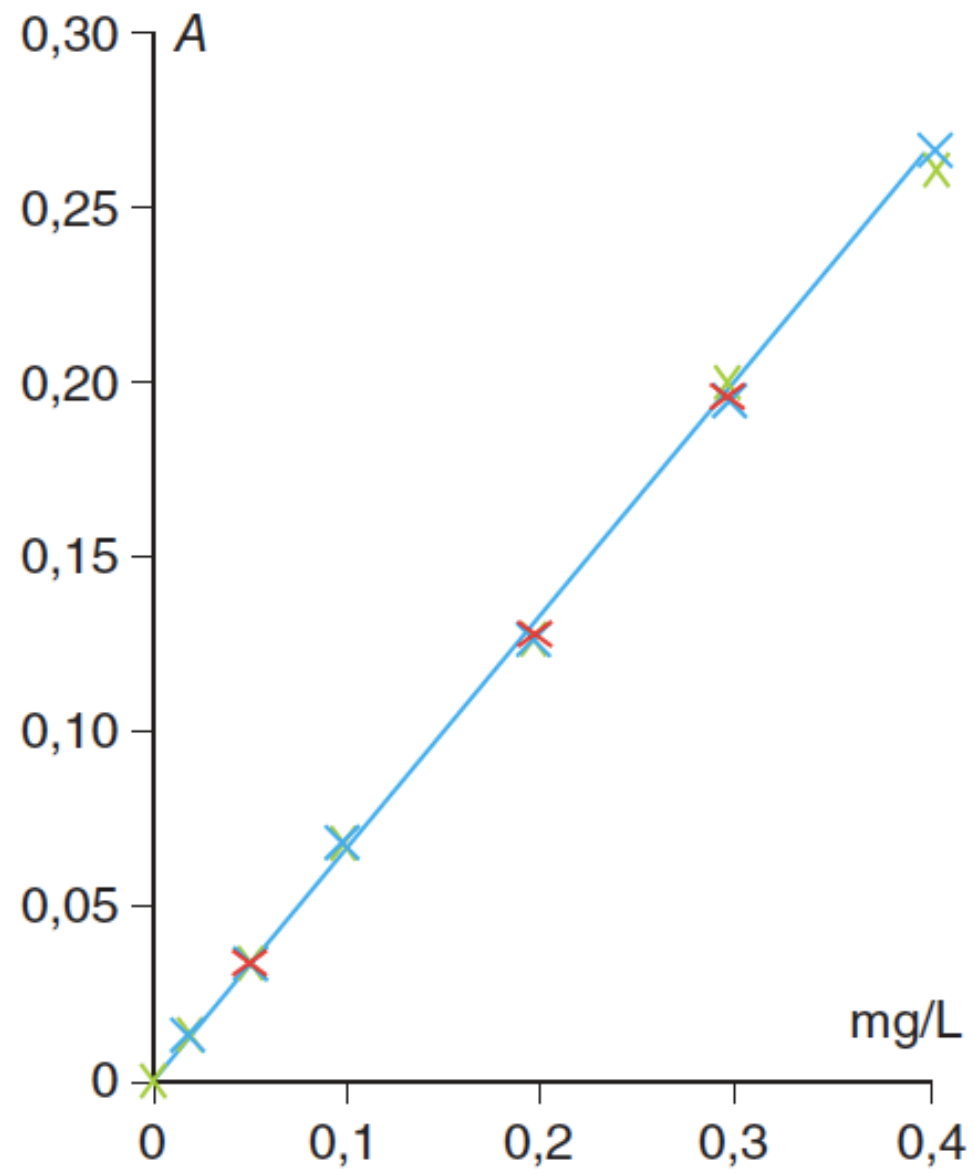
Rød streg: kurve med alle punkter

Grøn streg: kurve uden de to sidste punkter

Blå streg: kurve uden de sidste tre punkter



Figur 12.24. Linearitetsbestemmelse, tætte målinger.



Figur 12.25. Linearitet, AAS-metodevalidering.

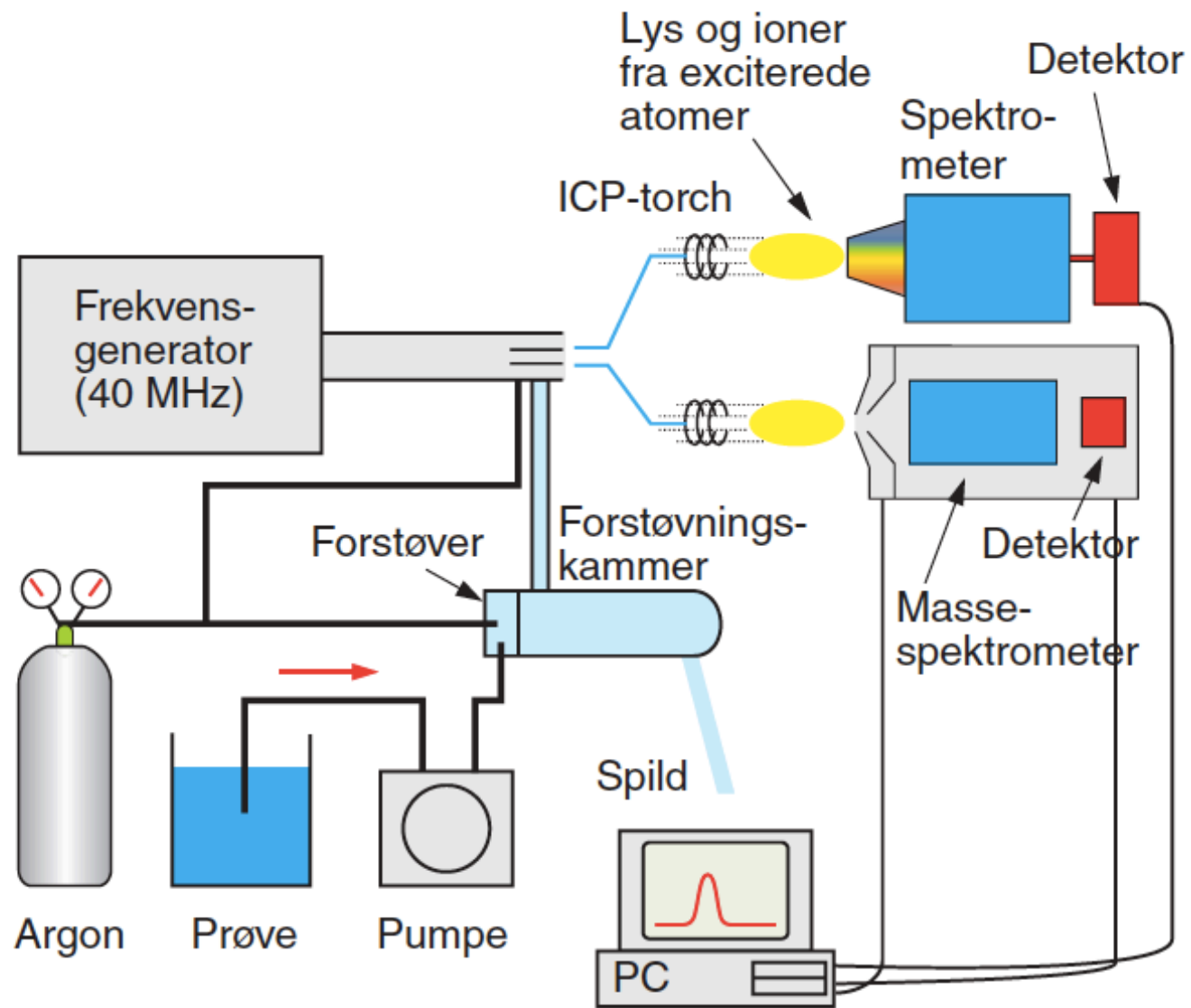
	ICP-MS	ICP-OES	Flamme-AAS	Grafitovns-AAS
Detektionsgrænse	ng/L*	µg/L	ng/L-µg/L	ng/L
Linært område**	10 ⁶	10 ⁵	10 ³	10 ²
Præcision:				
Kort tid	1-3 %	0,1-2 %	0,1-1 %	1-5 %
Lang tid (4 timer)	< 3 %	< 3 %	< 2 %	< 5 %
Antal elementer der kan bestemmes	> 75	> 73	> 68	> 50
Analysehastighed	Alle elementer 1-10 min/prøve	5-30 elementer 1-4 min/prøve	1 min/element	2-3 min/element
Prøvevolumen	0,1-2 mL/min	1-2 mL/min	5-10 mL/min	10 µL/prøve***
Metodeudvikling	Kræver træning	Kræver træning	Let	Kræver træning
Investering:				
Instrument (Kr.)	ca. 1 mio	ca. ½ mio	ca. 100 t	ca. 250 t
Driftsudgifter	+++	++	+	++

* detektionsgrænser ned til pg/L kan forekomme ved nye instrumenter

** i forhold til detektionsgrænsen

*** måles ikke kontinuert

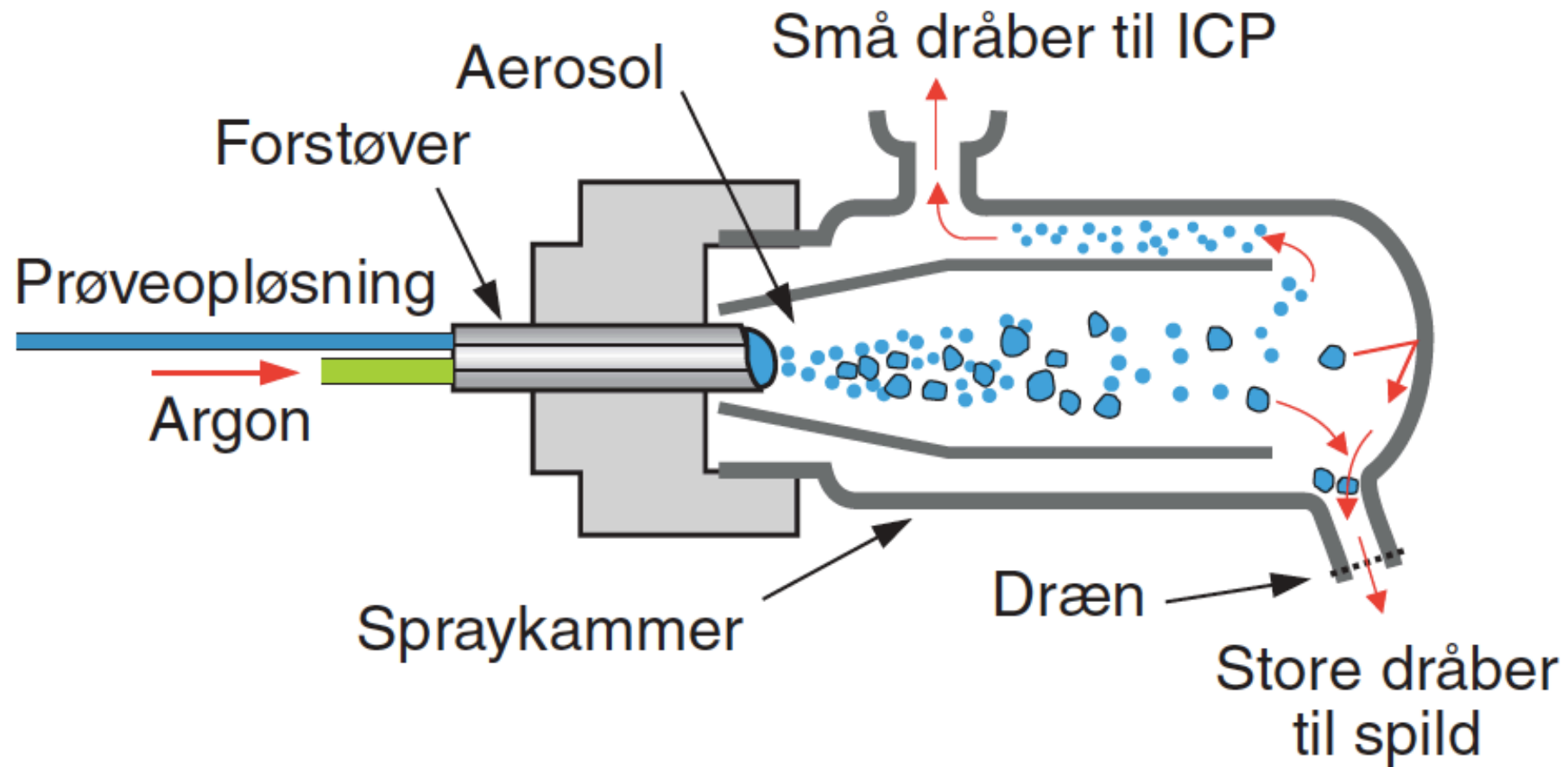
Tabel 13.1. Sammenligning af metoder til bestemmelse af metaller/grundstoffer.



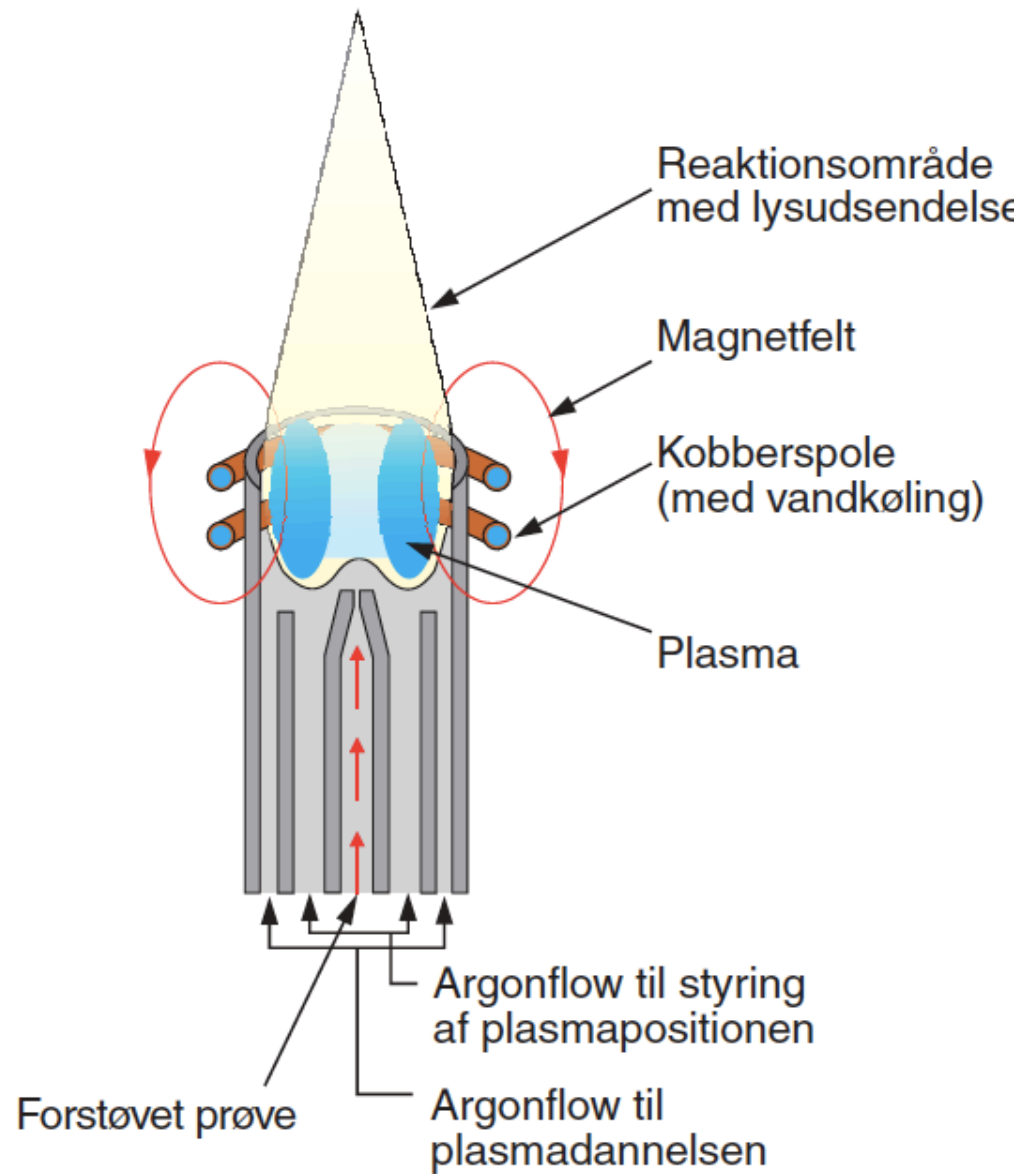
Figur 13.1. Principskitse af ICP-OES og ICP-MS.



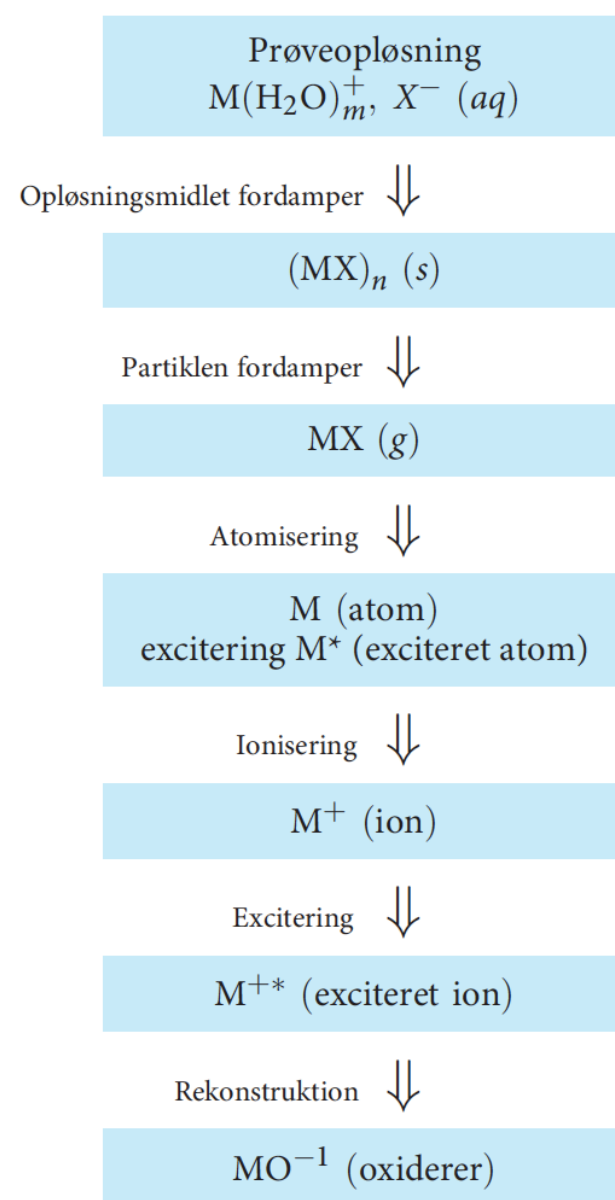
Figur 13.2. Koncentrisk forstøver.



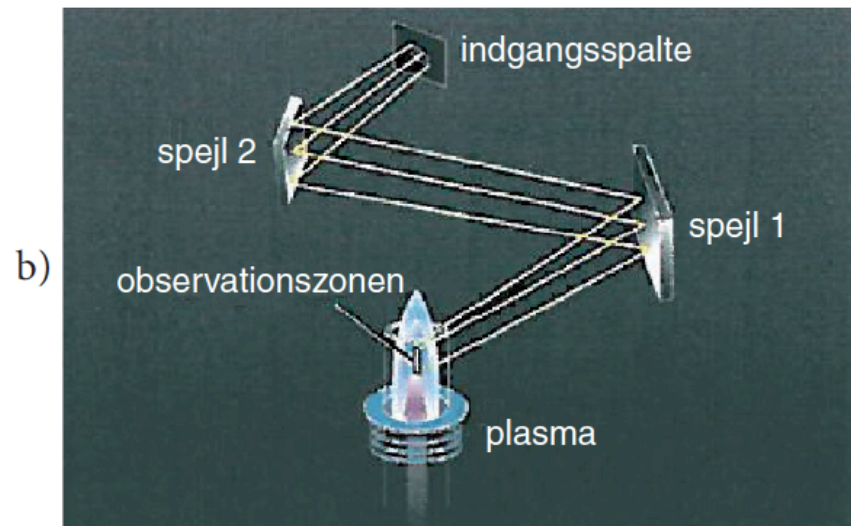
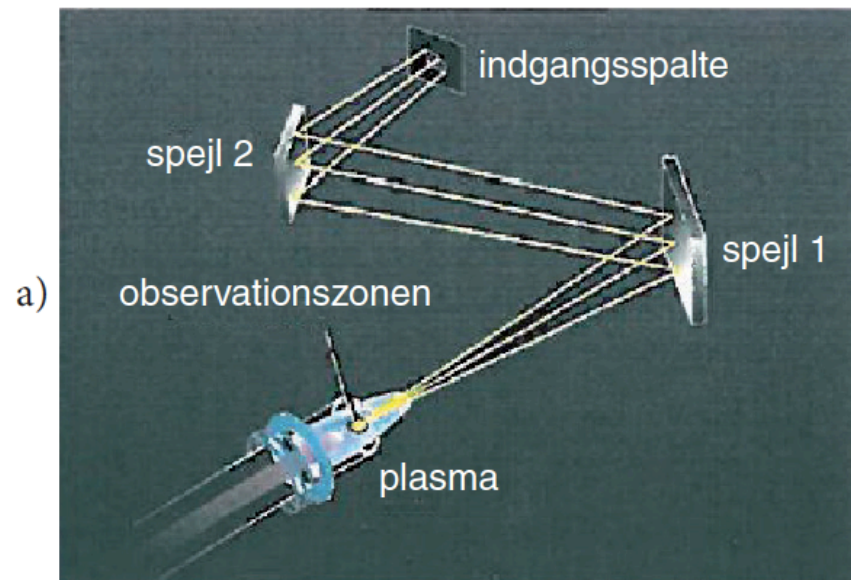
Figur 13.3. Forstøvningsproces i spraykammeret.



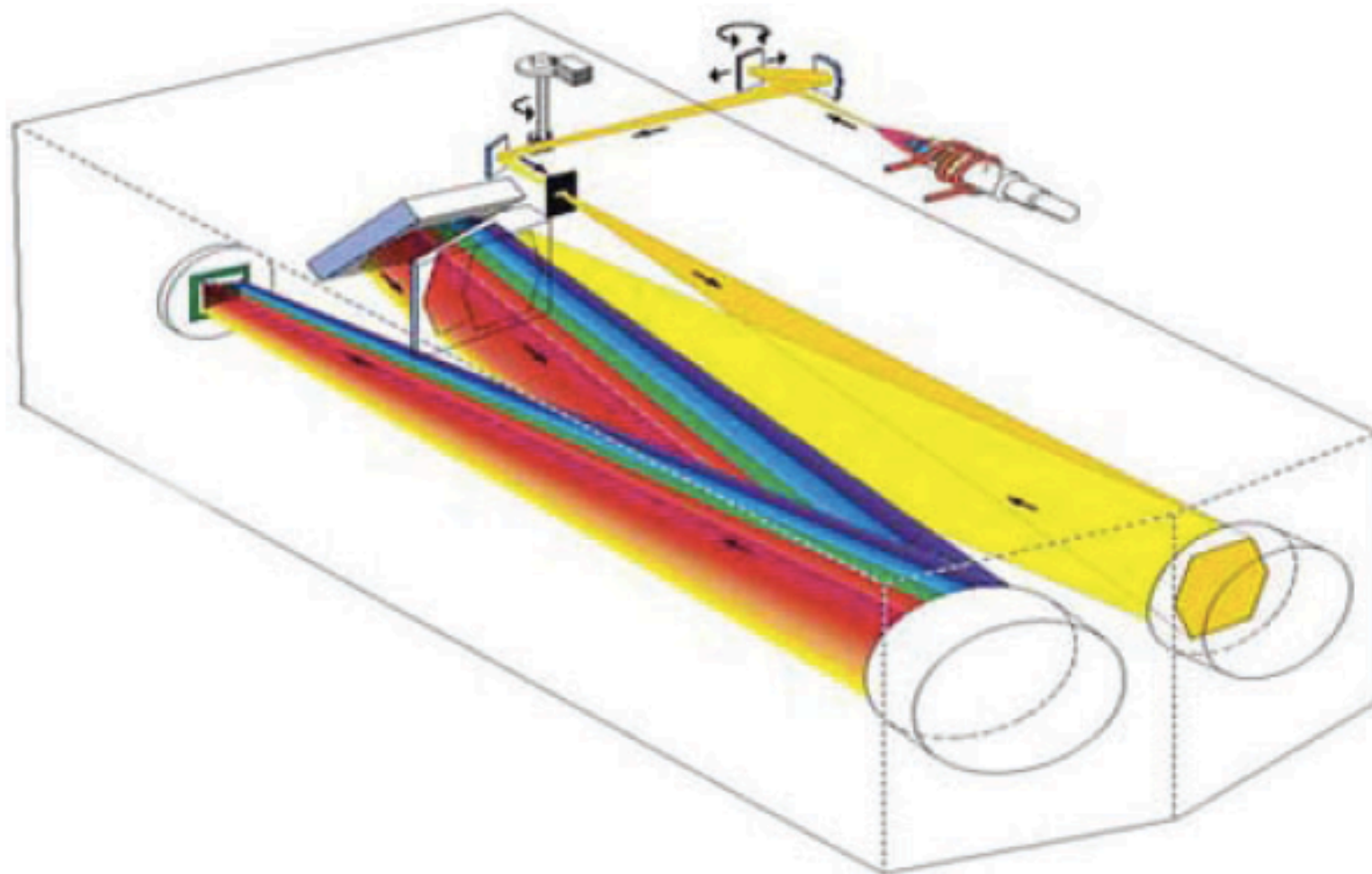
Figur 13.4. Torch med plasma og coil.



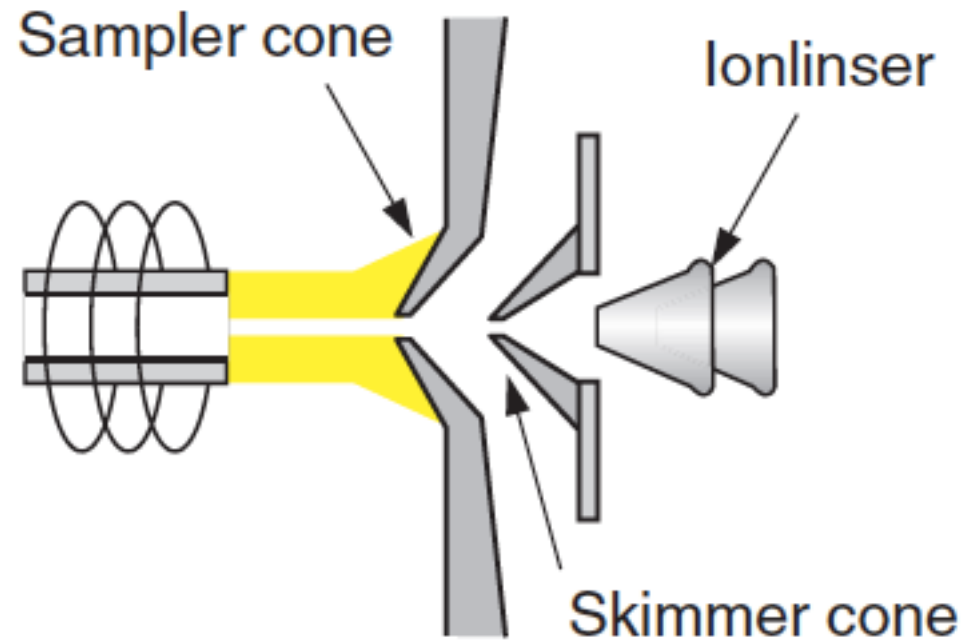
Figur 13.5. Processer i plasma.



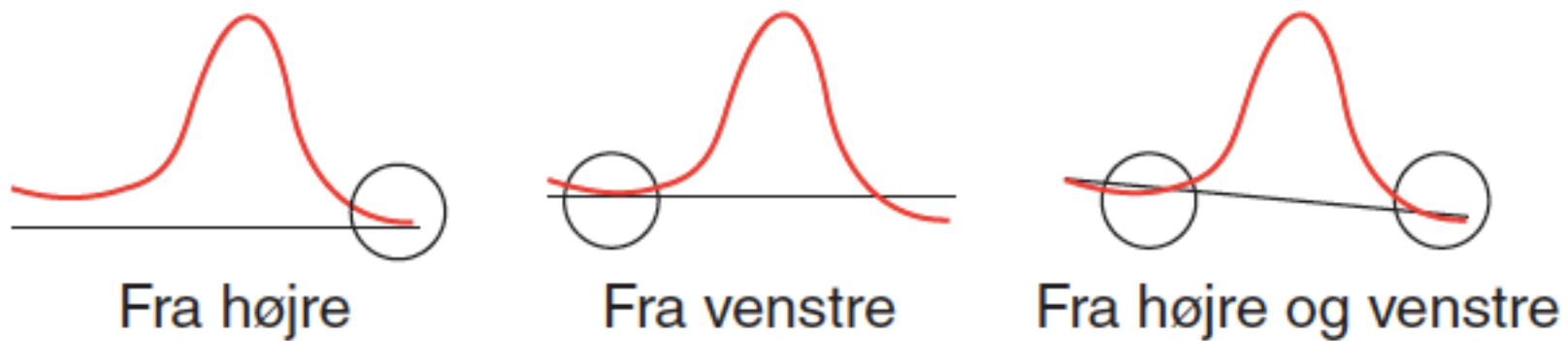
Figur 13.6. To forskellige strålegange mellem plasma og spektrometer a) aksialt og b) radialt.



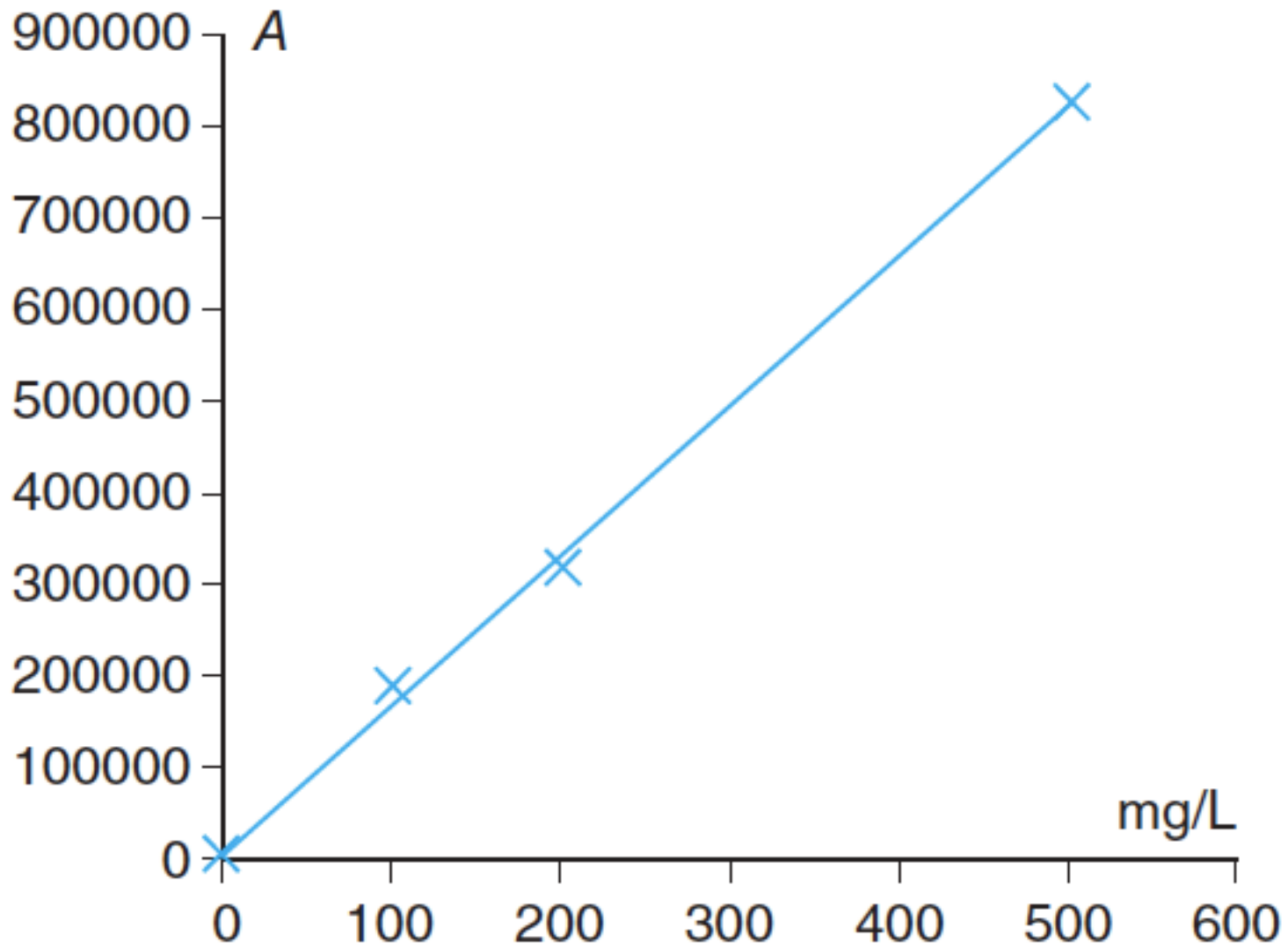
Figur 13.7. Torch, plasma, polykromator og detektor.



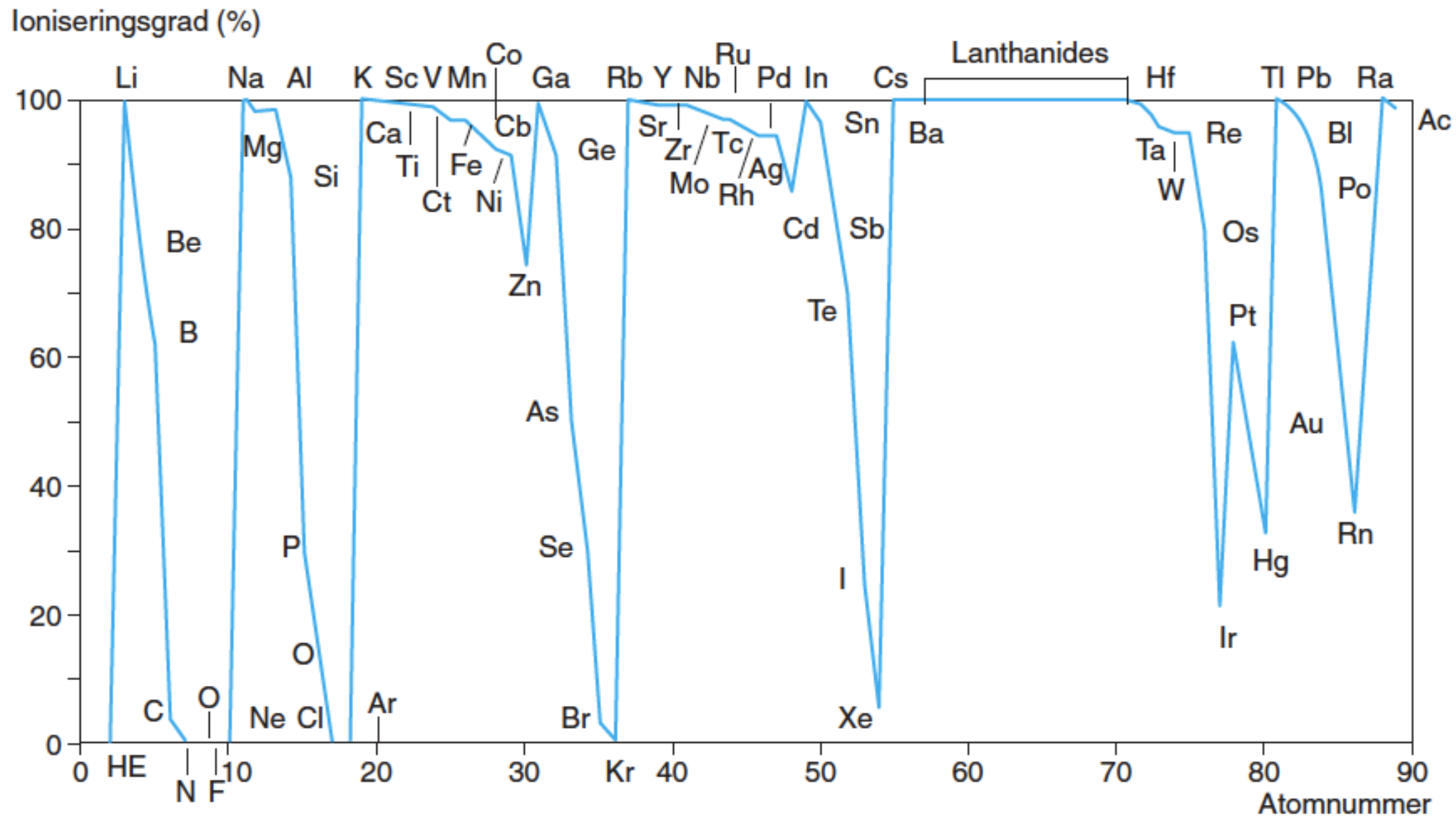
Figur 13.8. Interfaceregion.



Figur 13.9. Lineær baggrundskorrektion fra højre, venstre eller højre og venstre.



Figur 13.10.



Figur 13.12. Ioniseringsgrad i plasmaet for de forskellige grundstoffer ved 6850 °C.

	49	50	51	52	53	54	55	56
Ti	5,41	5,18						
V		0,250	99,75					
Cr		4,345		83,79	9,501	2,365		
Mn							100	
Fe						5,845		91,75
Co								

Figur 13.13. Udsnit af en isotopfordelingstabel.

Element	Polyatomiske interferenter	Monoatomiske interferenter
^{51}V	$^{35}\text{Cl}^{16}\text{O}$	
^{52}Cr	$^{40}\text{Ar}^{12}\text{C}$	
^{53}Cr	$^{37}\text{Cl}^{16}\text{O}$	
^{54}Fe		^{54}Cr
^{56}Fe	$^{40}\text{Ar}^{16}\text{O}$	
^{58}Ni	$^{42}\text{Ca}^{16}\text{O}$	^{58}Fe
^{60}Ni	$^{44}\text{Ca}^{16}\text{O}$	
^{63}Cu	$^{40}\text{Ar}^{23}\text{Na}$	
^{65}Cu	$^{40}\text{Ar}^{25}\text{Mg}$	
^{66}Zn	$^{34}\text{S}^{(16}\text{O})_2$	
^{68}Zn	$^{36}\text{Ba}^{+2}$	
^{75}As	$^{40}\text{Ar}^{35}\text{Cl}$	
^{80}Se	$^{40}\text{Ar}^{40}\text{Ar}$	

Tabel 13.2. Mest betydningsfulde interferenter.

Element/ Isotop	Matrix/ Solvent	Interference
^{39}K	H_2O	^{38}ArH
^{40}Ca	H_2O	^{40}Ar
^{56}Fe	H_2O	$^{40}\text{Ar}^{36}\text{O}$
^{80}Se	H_2O	$^{40}\text{Ar}^{16}\text{Ar}$
^{51}V	HCl	$^{35}\text{Cl}^{16}\text{O}$
^{75}As	HCl	$^{40}\text{Ar}^{35}\text{Cl}$
^{29}Si	HNO_3	$^{14}\text{N}^{14}\text{N}$
^{44}Ca	HNO_3	$^{14}\text{N}^{14}\text{N}^{16}\text{O}$
^{56}Mn	HNO_3	$^{40}\text{Ar}^{15}\text{N}$
^{48}Ti	H_2SO_4	$^{32}\text{S}^{16}\text{O}$
^{52}Cr	H_2SO_4	$^{34}\text{S}^{18}\text{O}$
^{64}Zn	H_2SO_4	$^{32}\text{S}^{16}\text{O}^{16}\text{O}$
^{63}Cu	H_3PO_4	$^{31}\text{P}^{16}\text{O}^{16}\text{O}$
^{24}Mg	Organics	$^{12}\text{C}^{12}\text{C}$
^{52}Cr	Organics	$^{40}\text{Ar}^{32}\text{C}$
^{65}Cu	Minerals	$^{48}\text{Ca}^{16}\text{OH}$
^{64}Zn	Minerals	$^{48}\text{Ca}^{16}\text{O}$
^{63}Cu	Seawater	$^{40}\text{Ar}^{23}\text{Na}$

Tabel 13.3. Vigtige interferenter fra forskellige matricer (fra Spectroscopy 17 (10), October 2002).