# Synchronous gas electron diffraction and mass-spectrometric experiments in Bielefeld

Yury V. Vishnevskiy

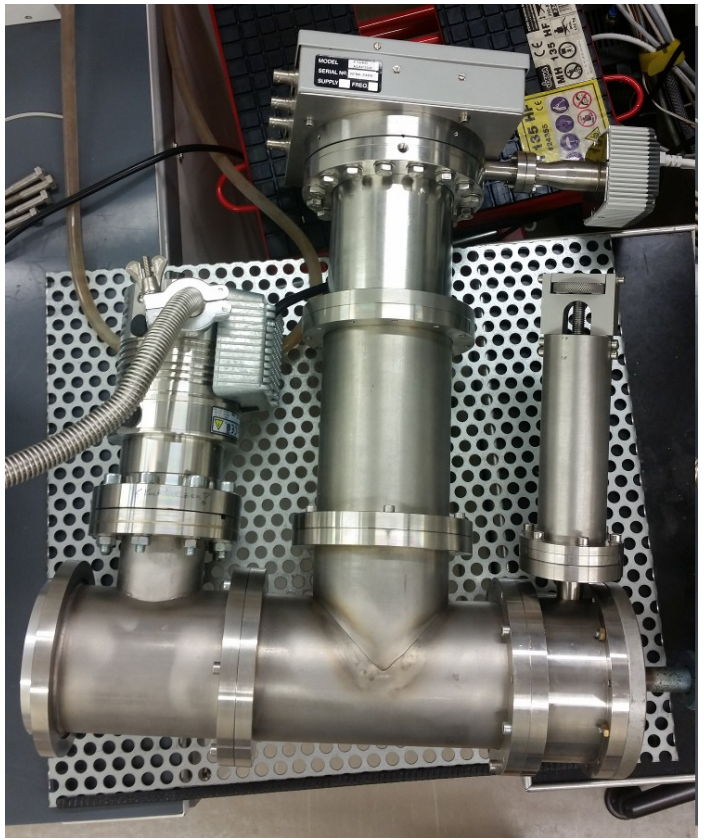
18th ESGED, Hirschegg, Kleinwalsertal, Austria, June 30th – July 4th, 2019

# The Instrument

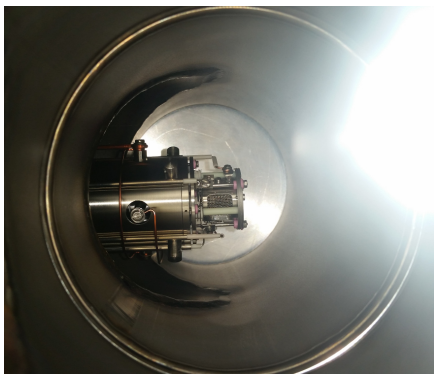
# 2015

# New parts

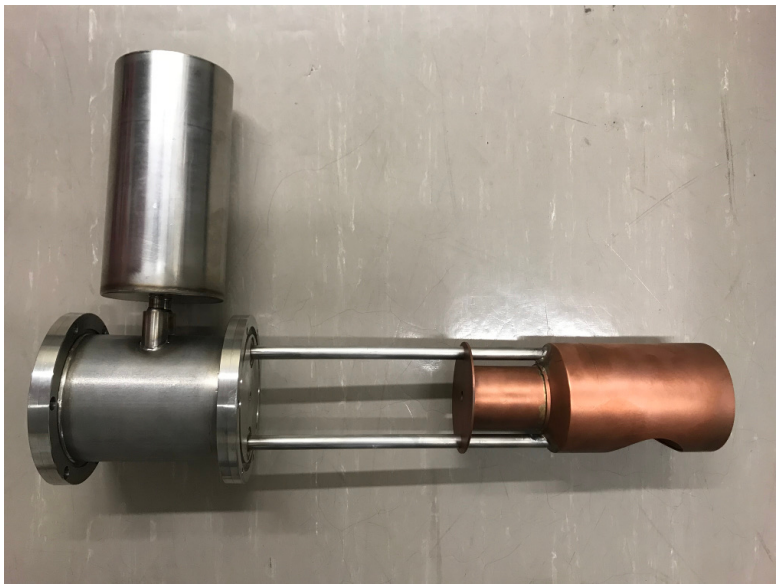
MS unit



EI



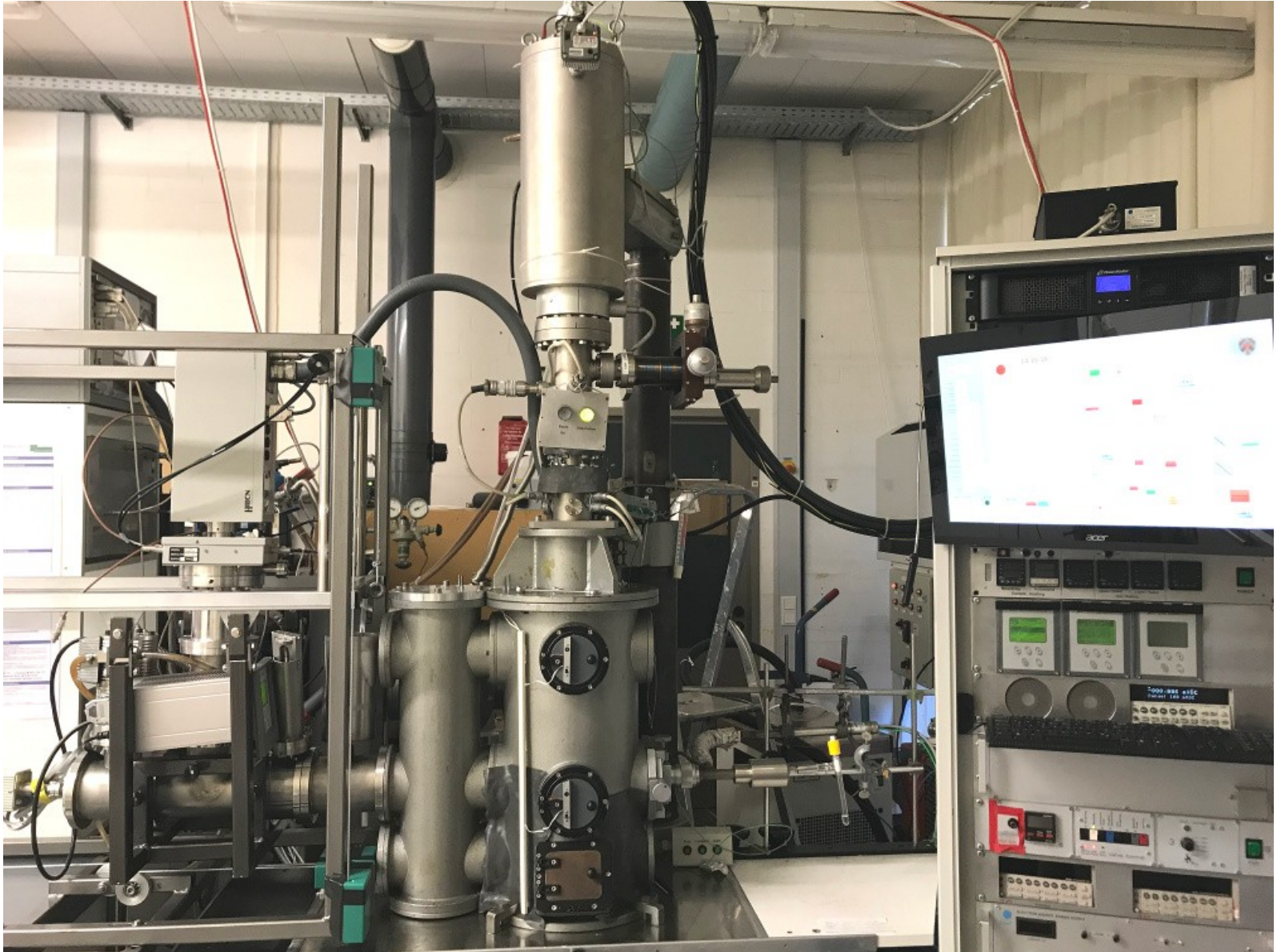
Cold trap



# October 2018

# MS unit in frame

EDWARDS
Norm
Back
Off
AIGX 2.39 x10 -07
Gge2 Not Connected
Gge3 Not Connected
mbar Rly 1 2 3

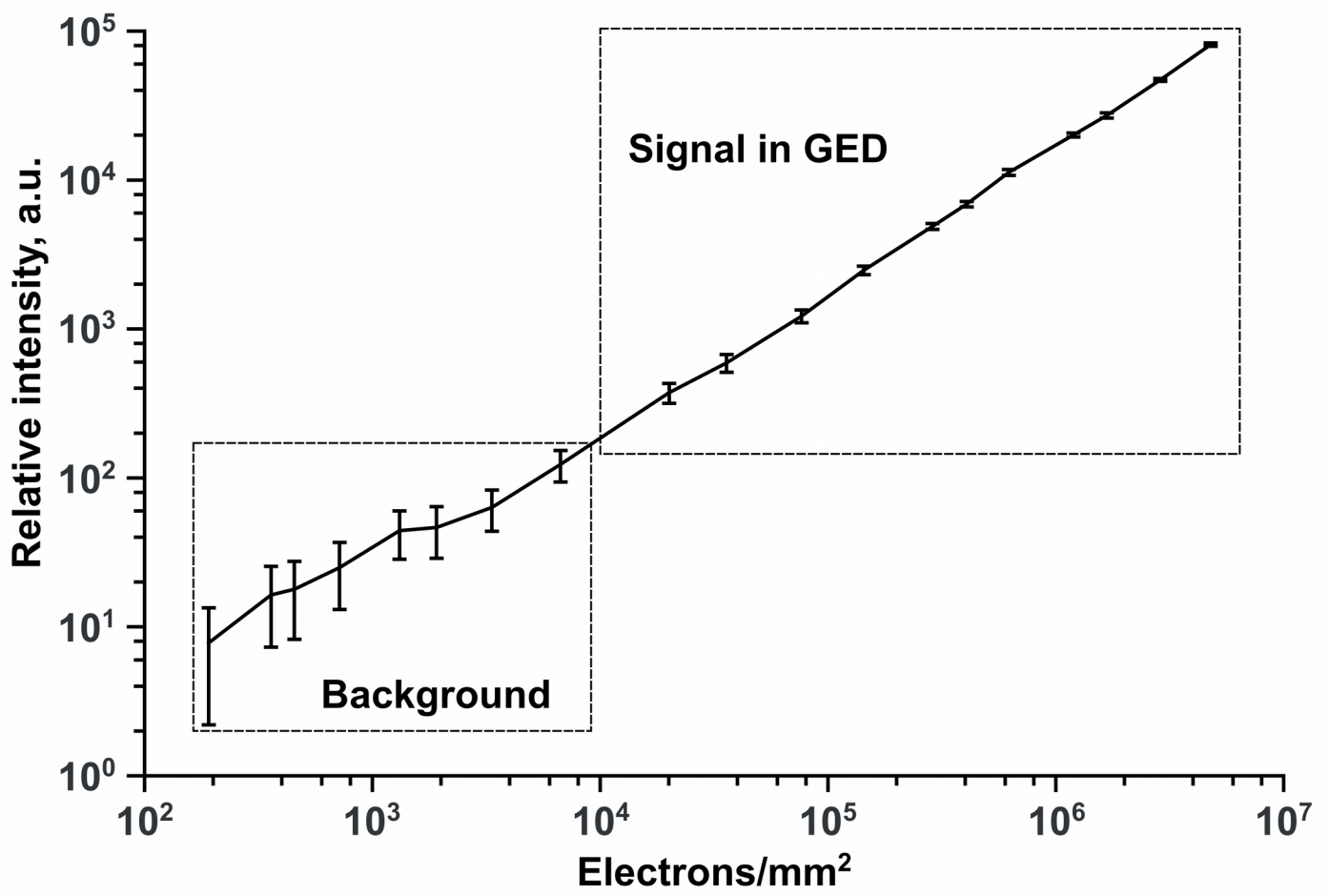


# Detector

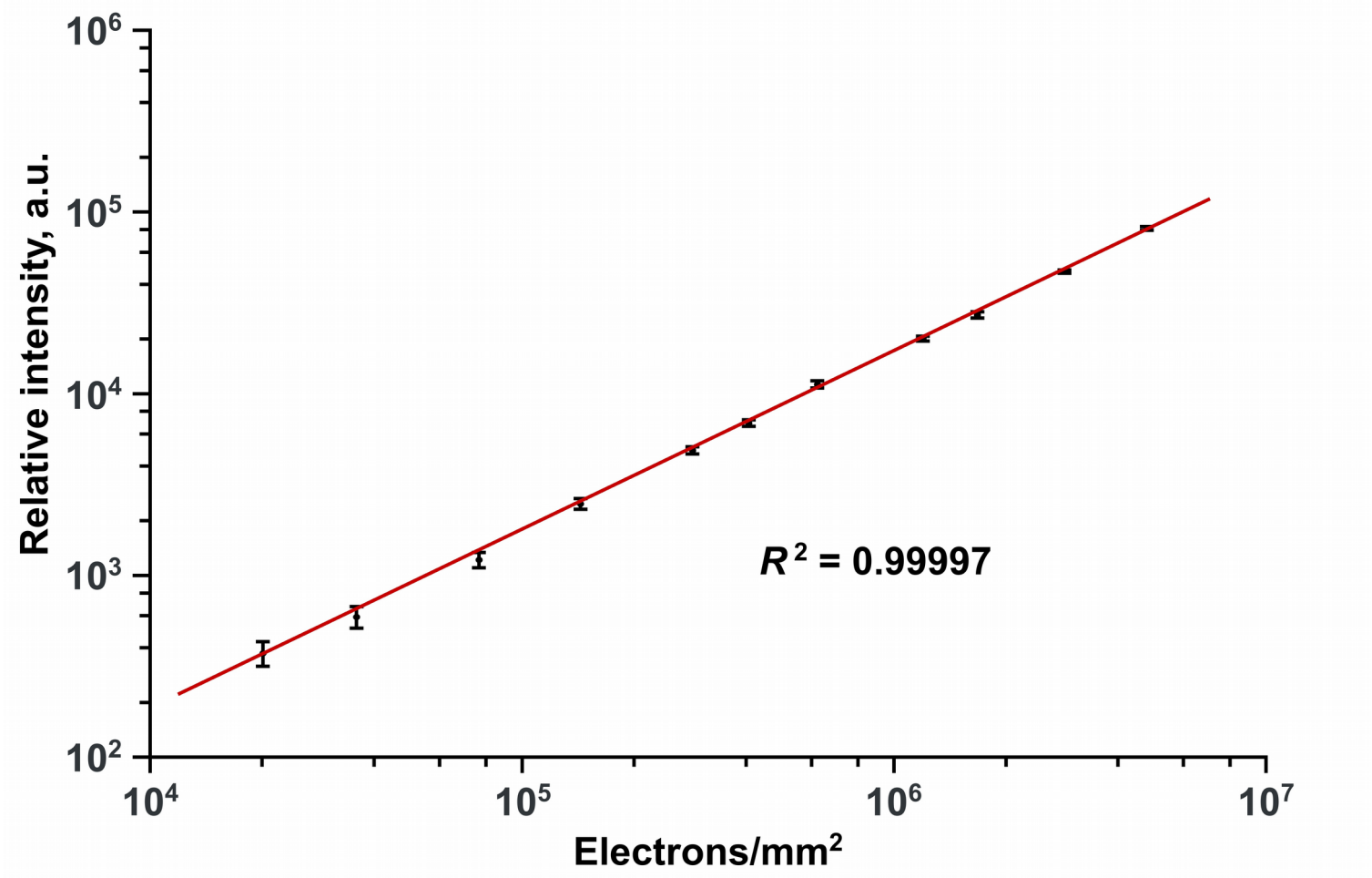
# Old Fuji BAS-1800II

# New Amersham Typhoon

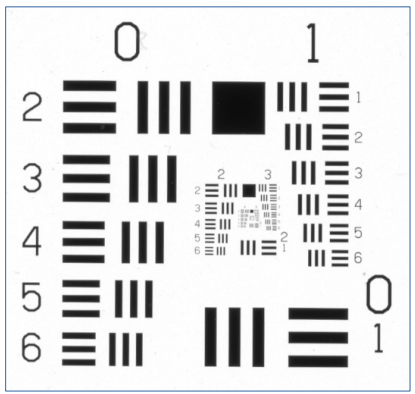
# Calibration with $^{14}C$

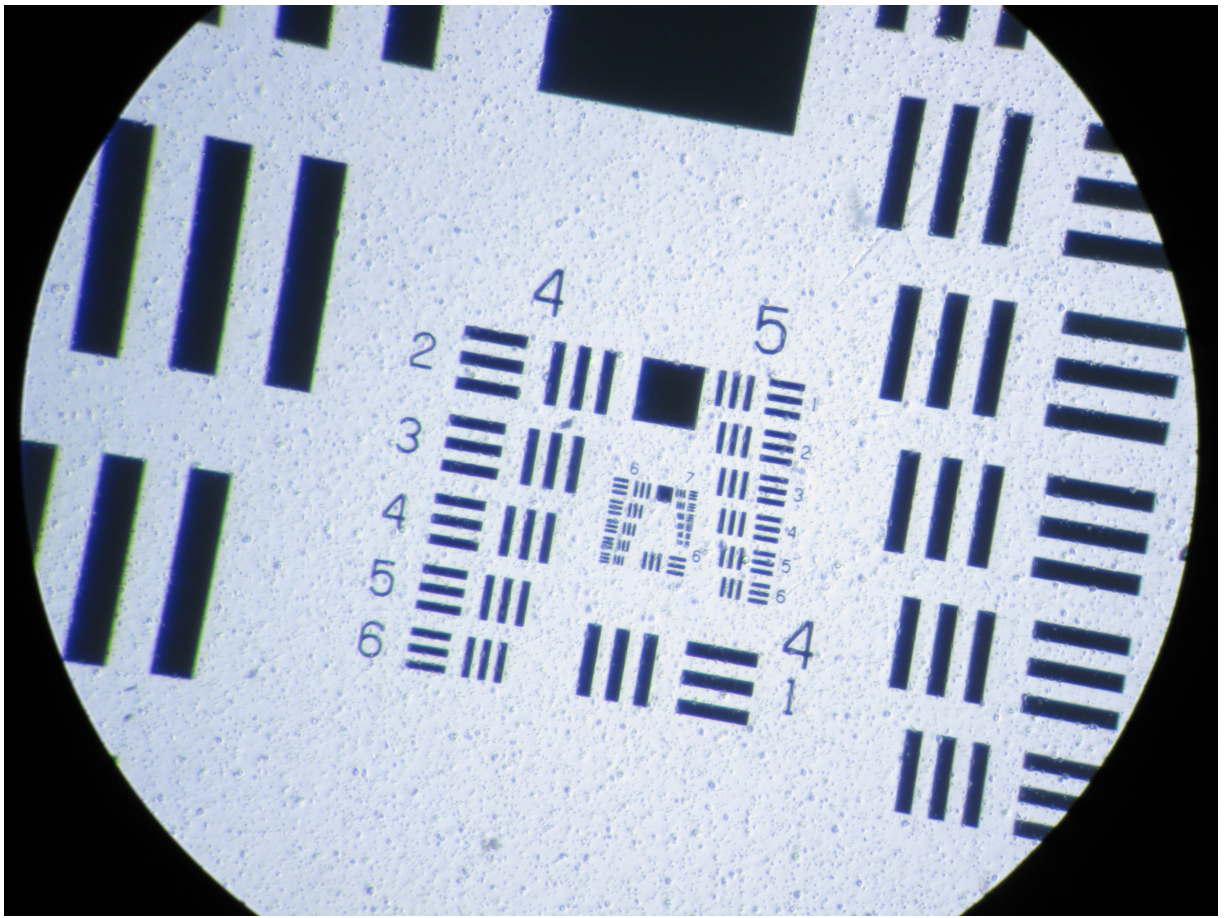


# Calibration with $^{14}C$

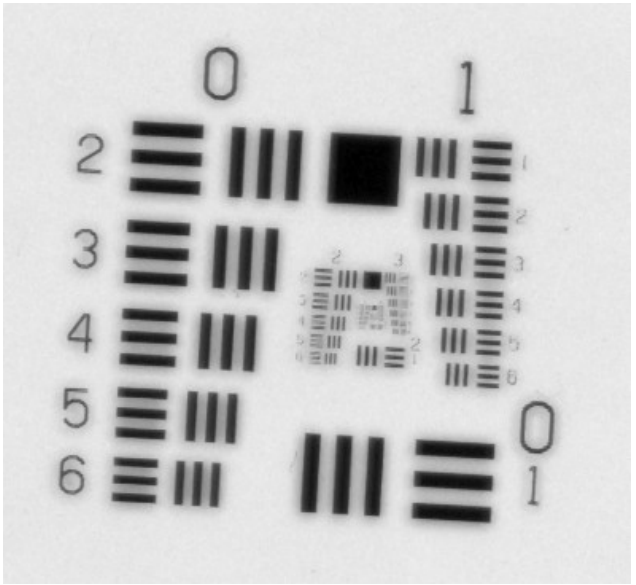


# Calibration with USAF-1951





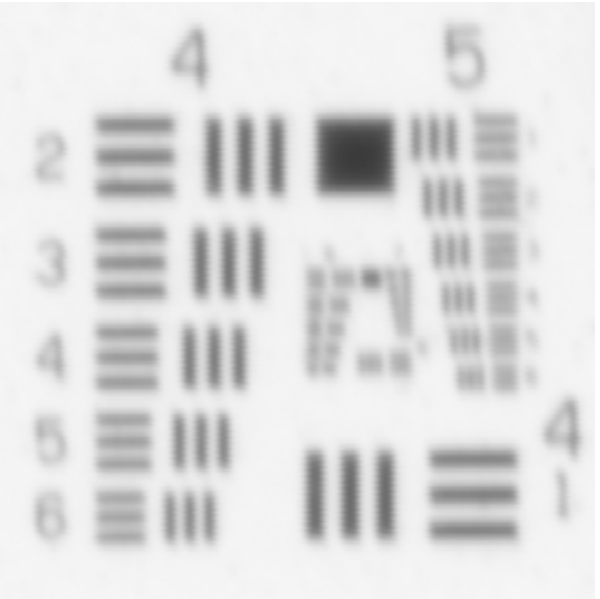
Typhoon
in 0.05 mm (**508** dpi) OD mode:




Determined resolution
at least **362** dpi but lt. **406** dpi

Element 0-2: **2.24**(**7**) mm,
expected **2.23** mm

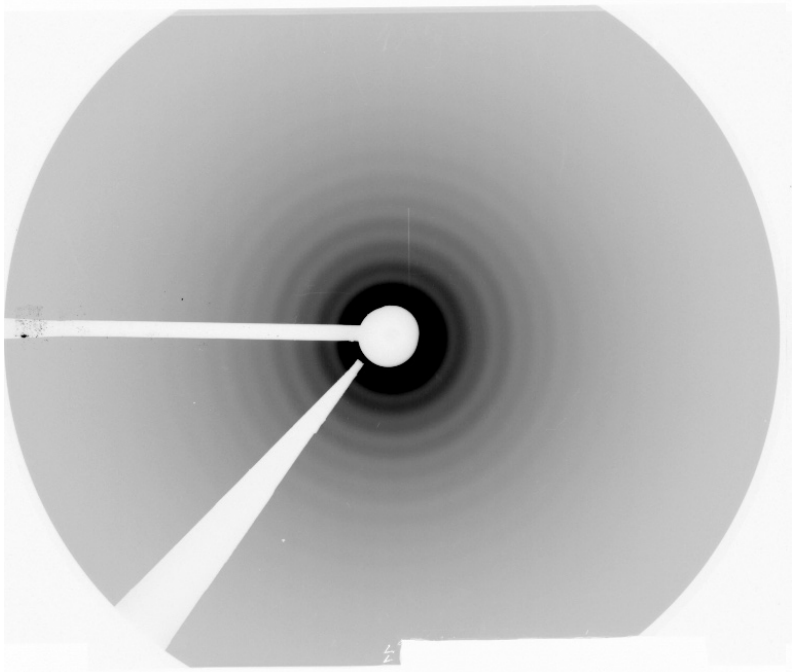
EPSON Perfection V850 Pro
in **6400** dpi mode:

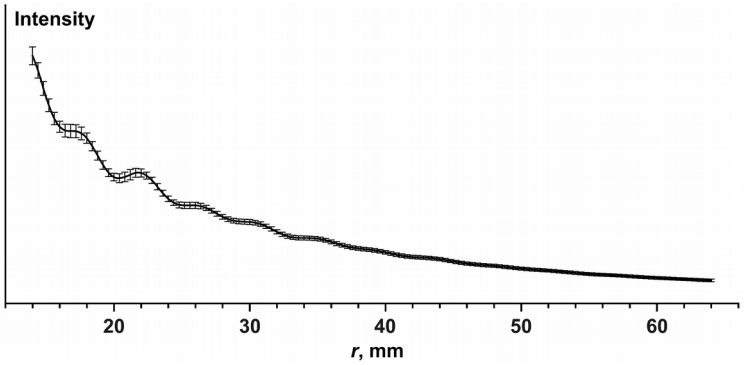


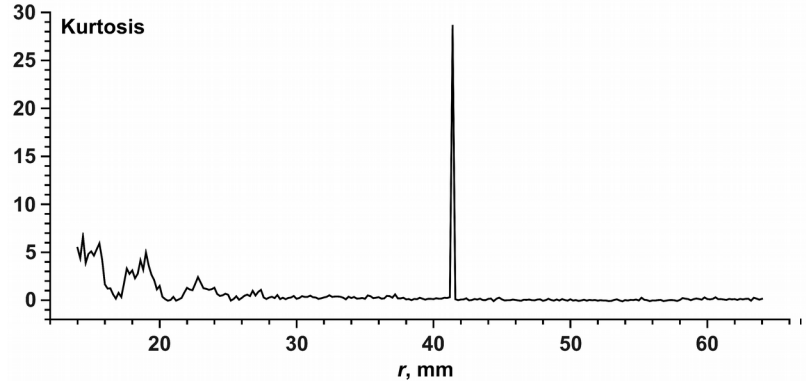

Determined resolution
at least **2048** dpi but lt. **2580** dpi

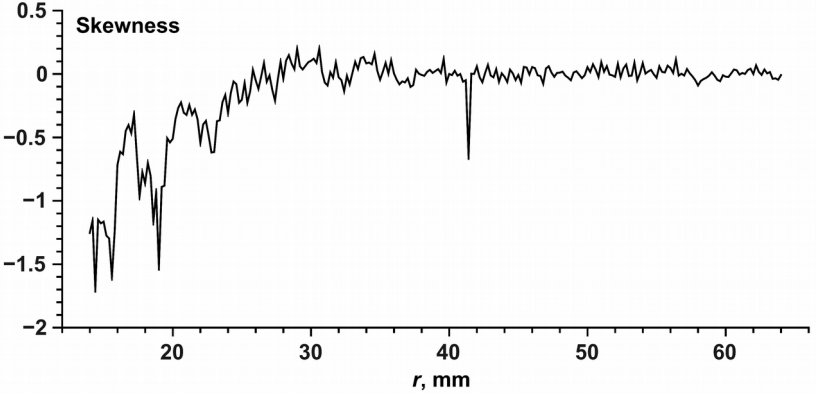
# Data reduction

Signal coding:
IMG, exp (like in Fuji BAS)
GEL, sqrt
TIF, linear





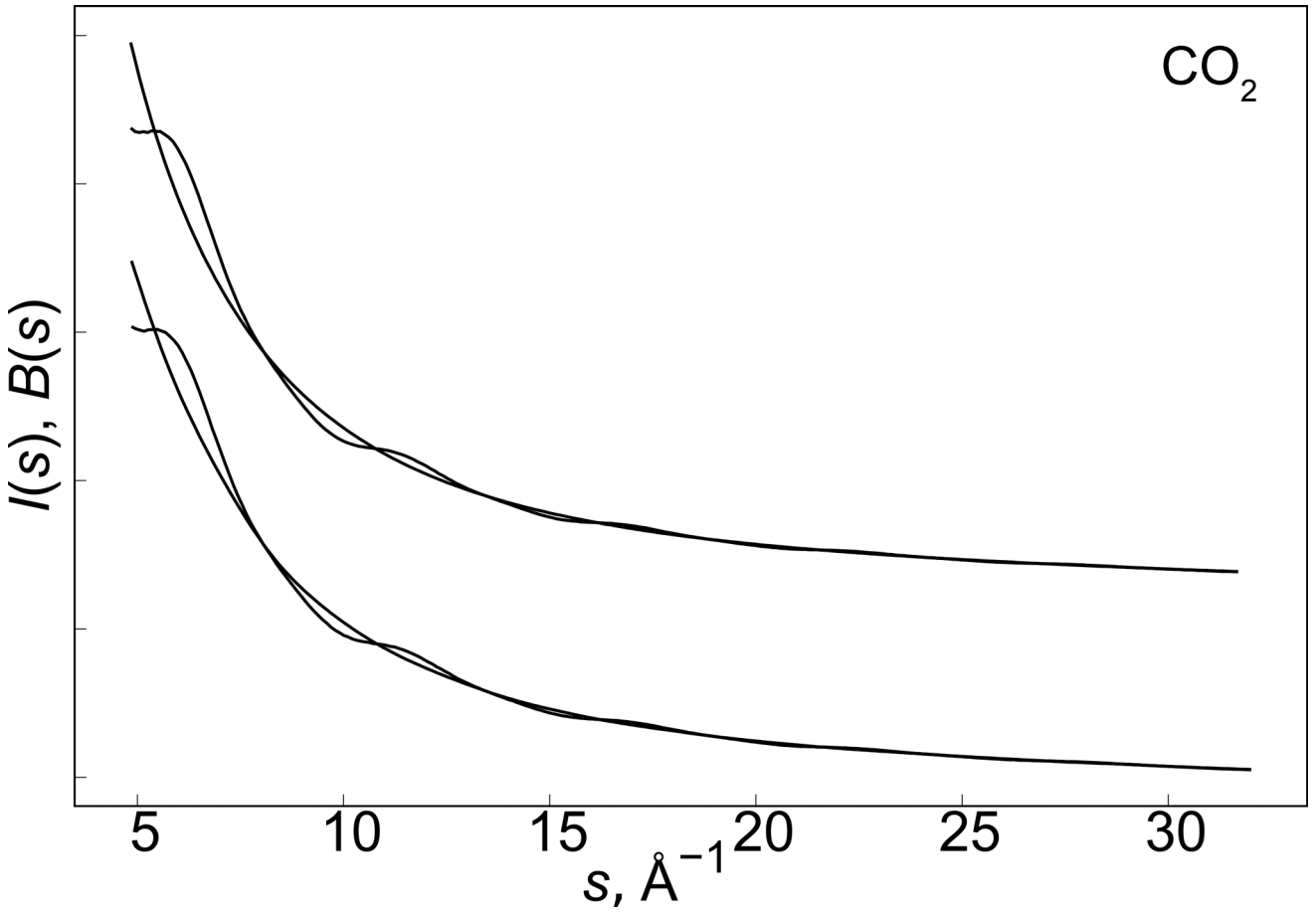




New module in UNEX2 for data reduction.

# Background

Background smoothness?
2nd derivatives?
Inflection points?

$$sM(s) = \frac{I(s) - B(s)}{B(s)} s$$

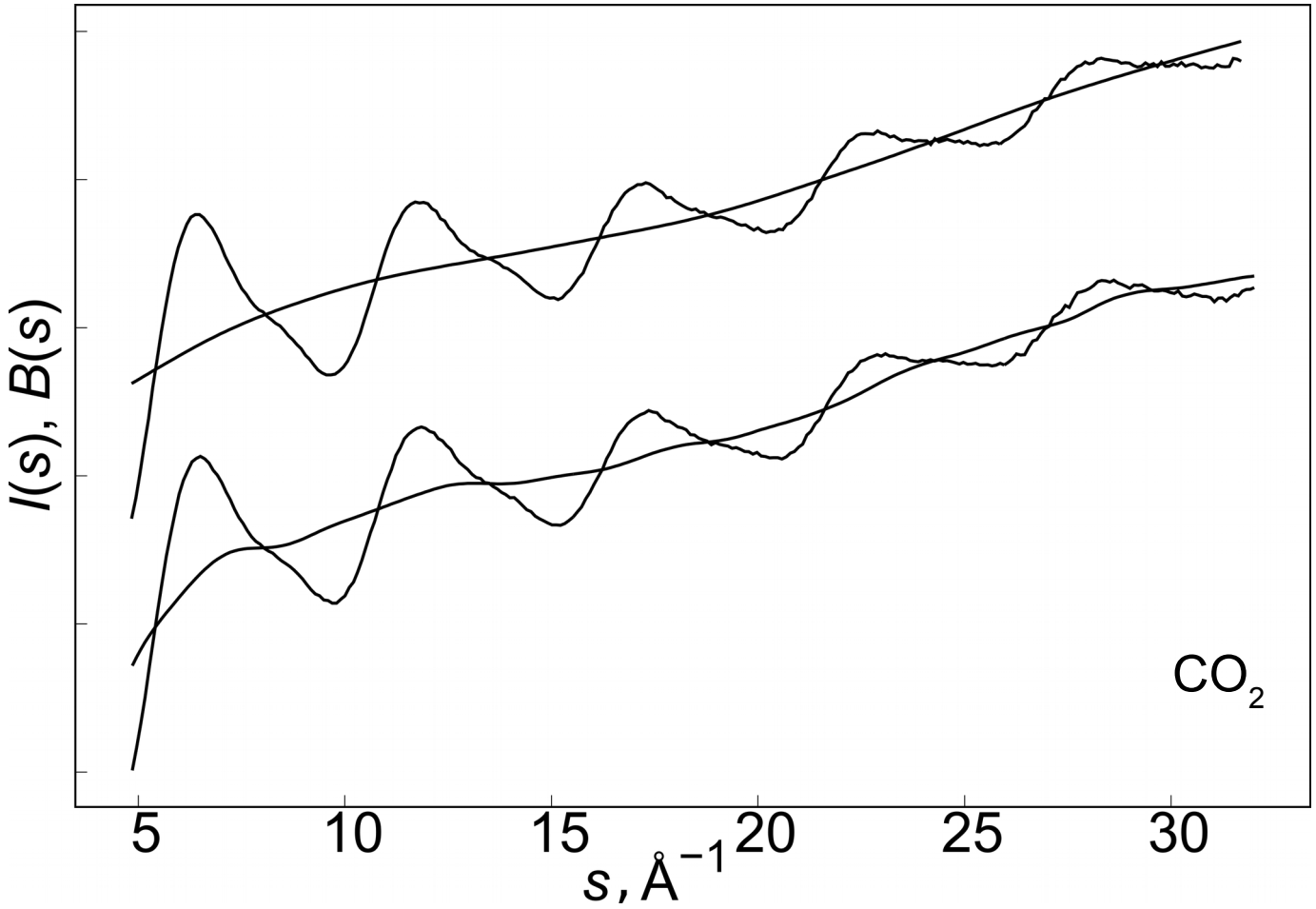


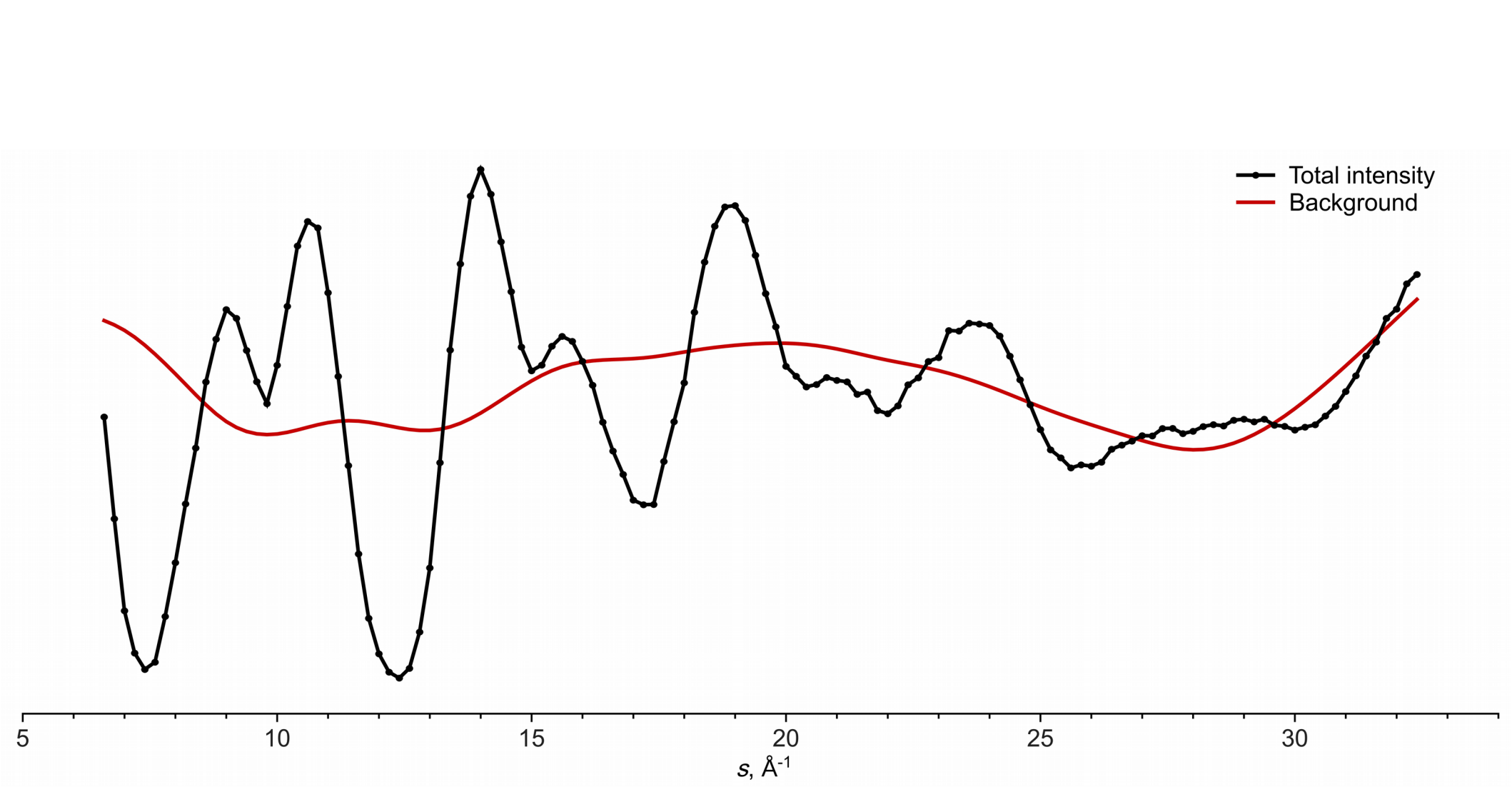
# Reduced intensity and background

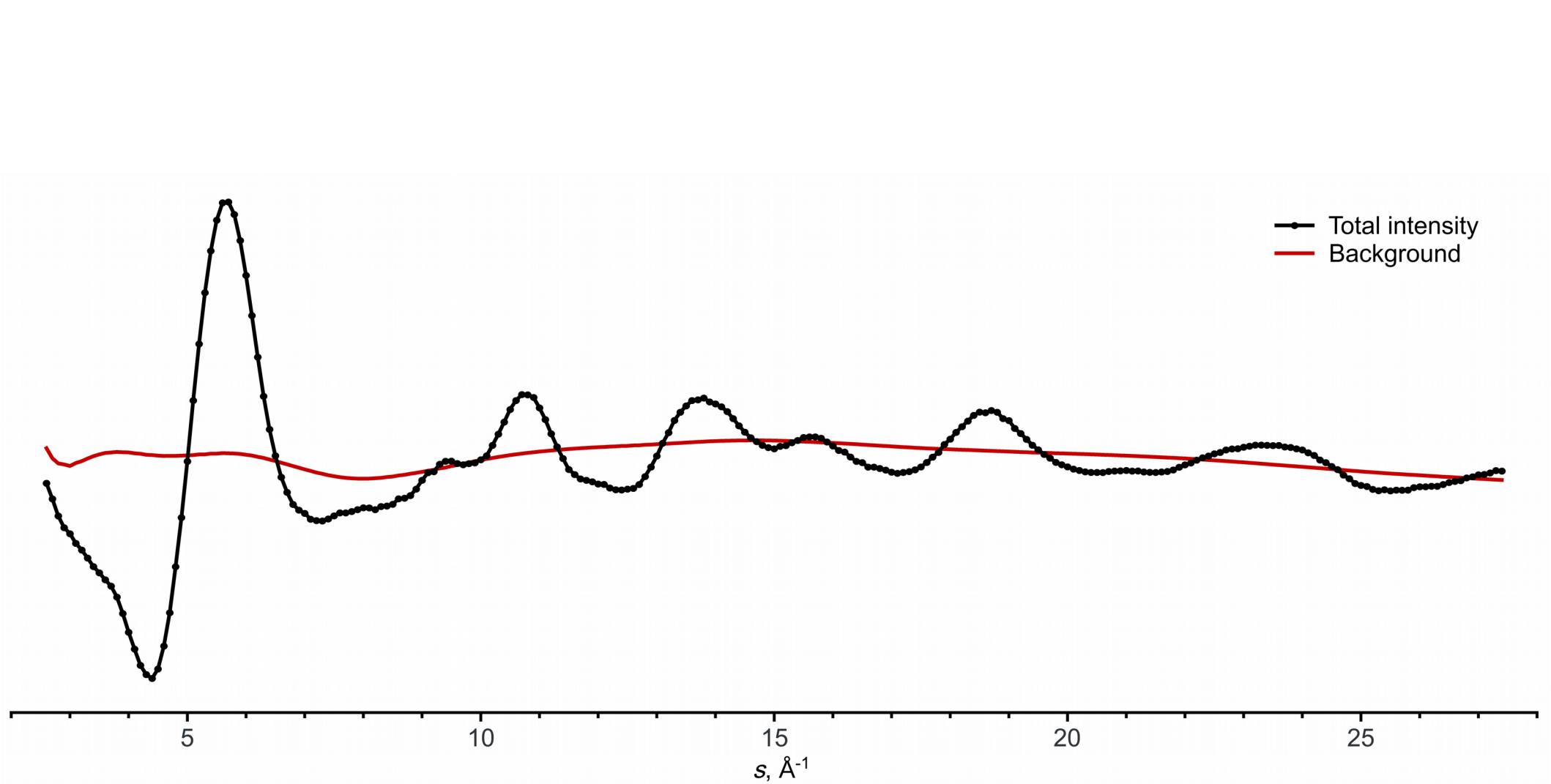
$$sM(s)=\frac{I(s)-B(s)}{B(s)}s$$

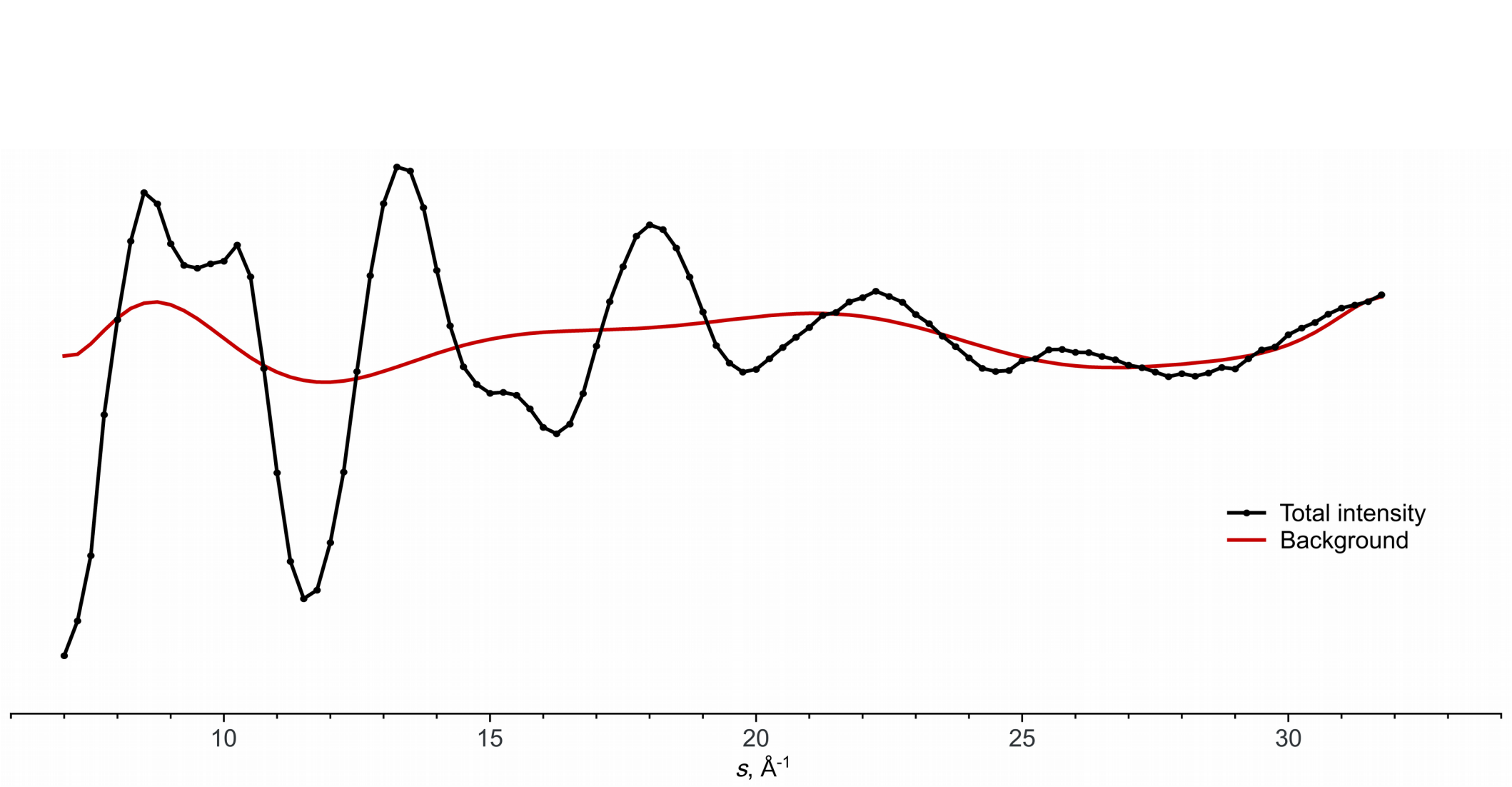
Reduced $I(s)$, $B(s)$:

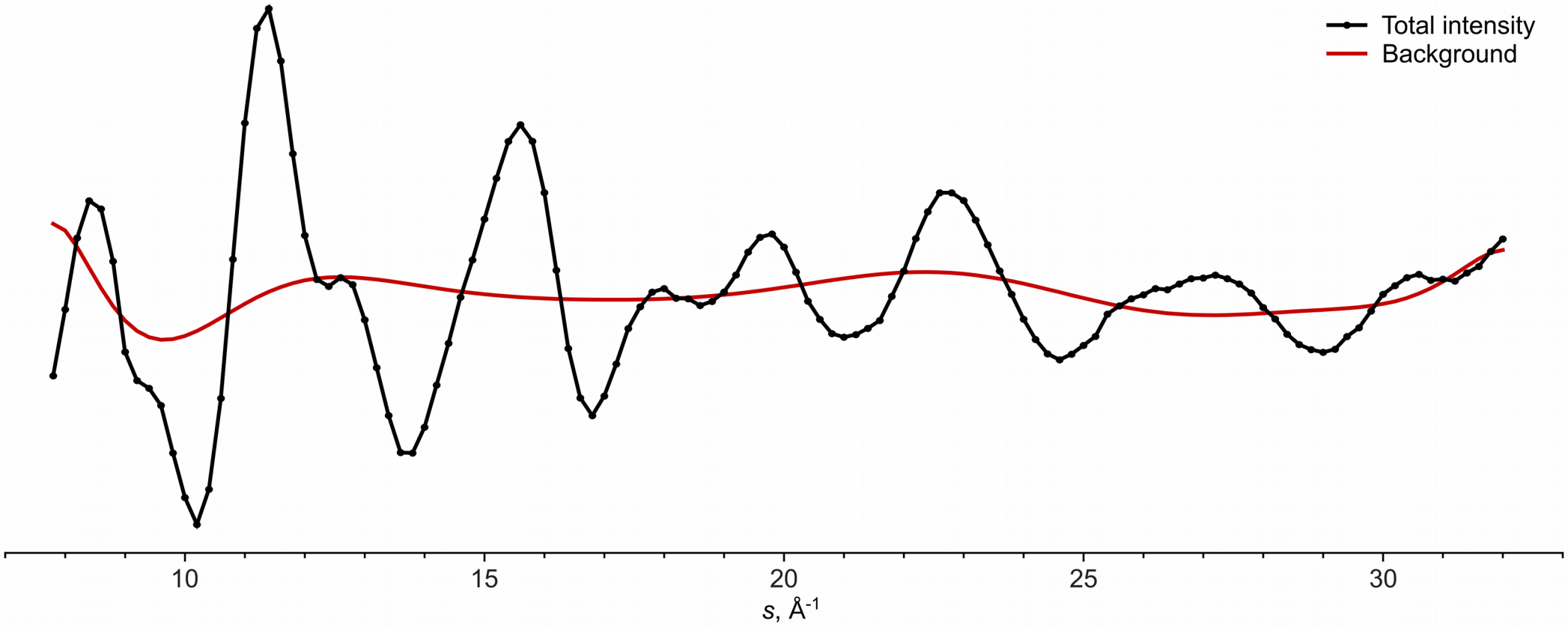
$$\frac{I(s)}{I_{at}(s)K} \qquad \frac{B(s)}{I_{at}(s)K}$$




Total intensity
Background
5
10
15
20
25
30
s, Å-1

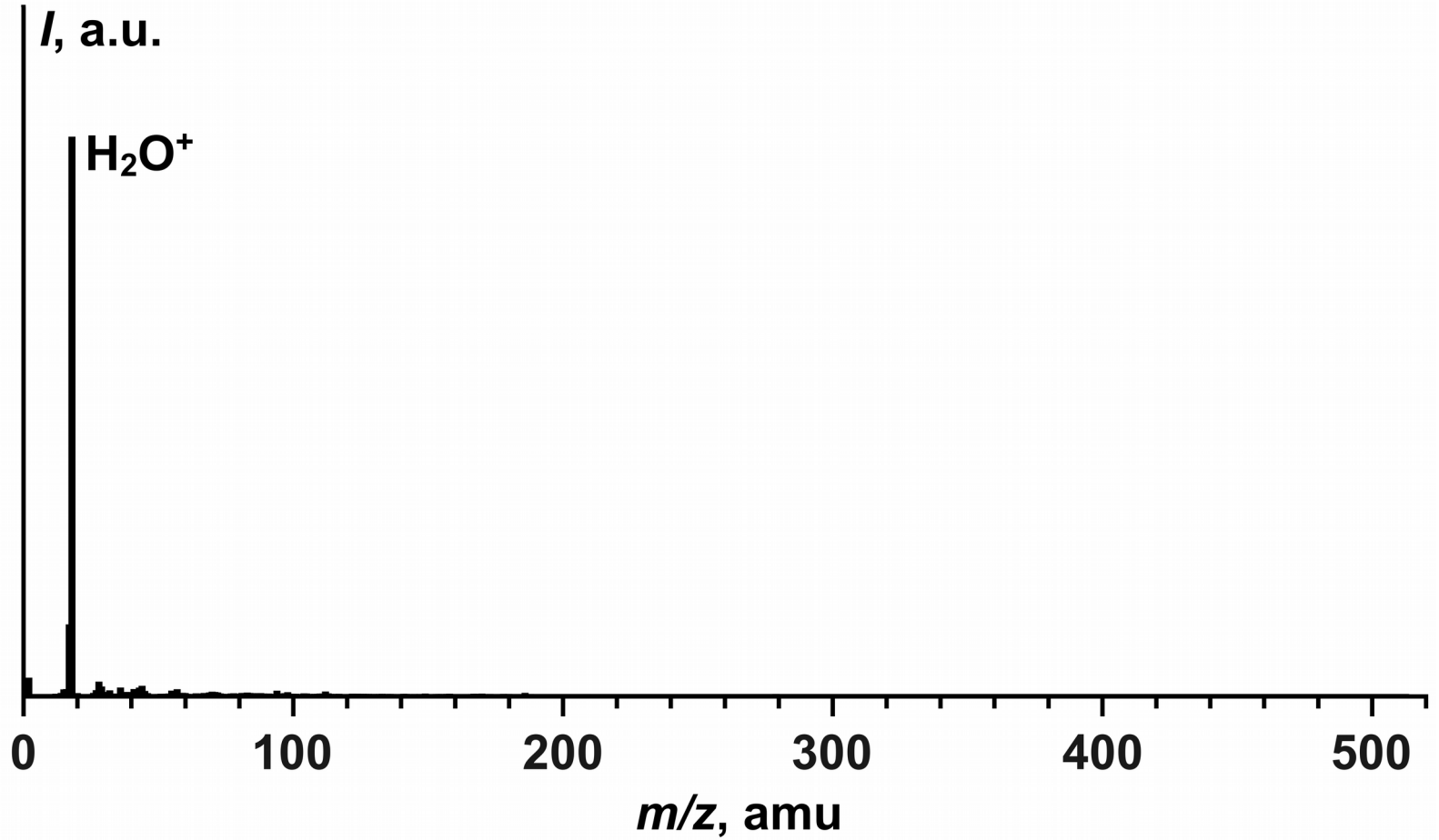

Total intensity
Background
5
10
15
20
25
s, Å-1


Total intensity
Background
10
15
20
25
30
s, Å-1

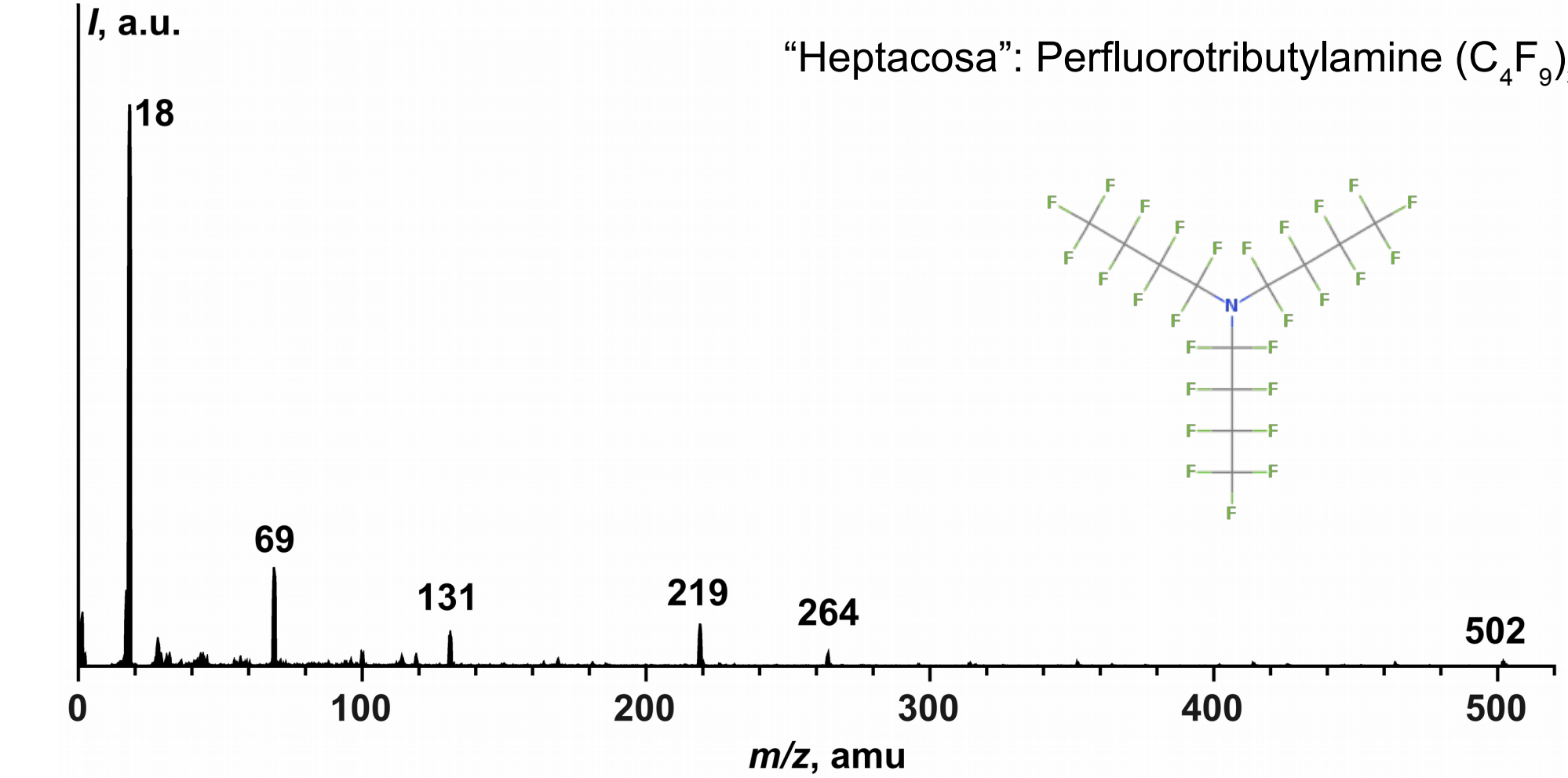

Total intensity
Background
10
15
20
25
30
s, Å-1

# Mass-spectra

# Background

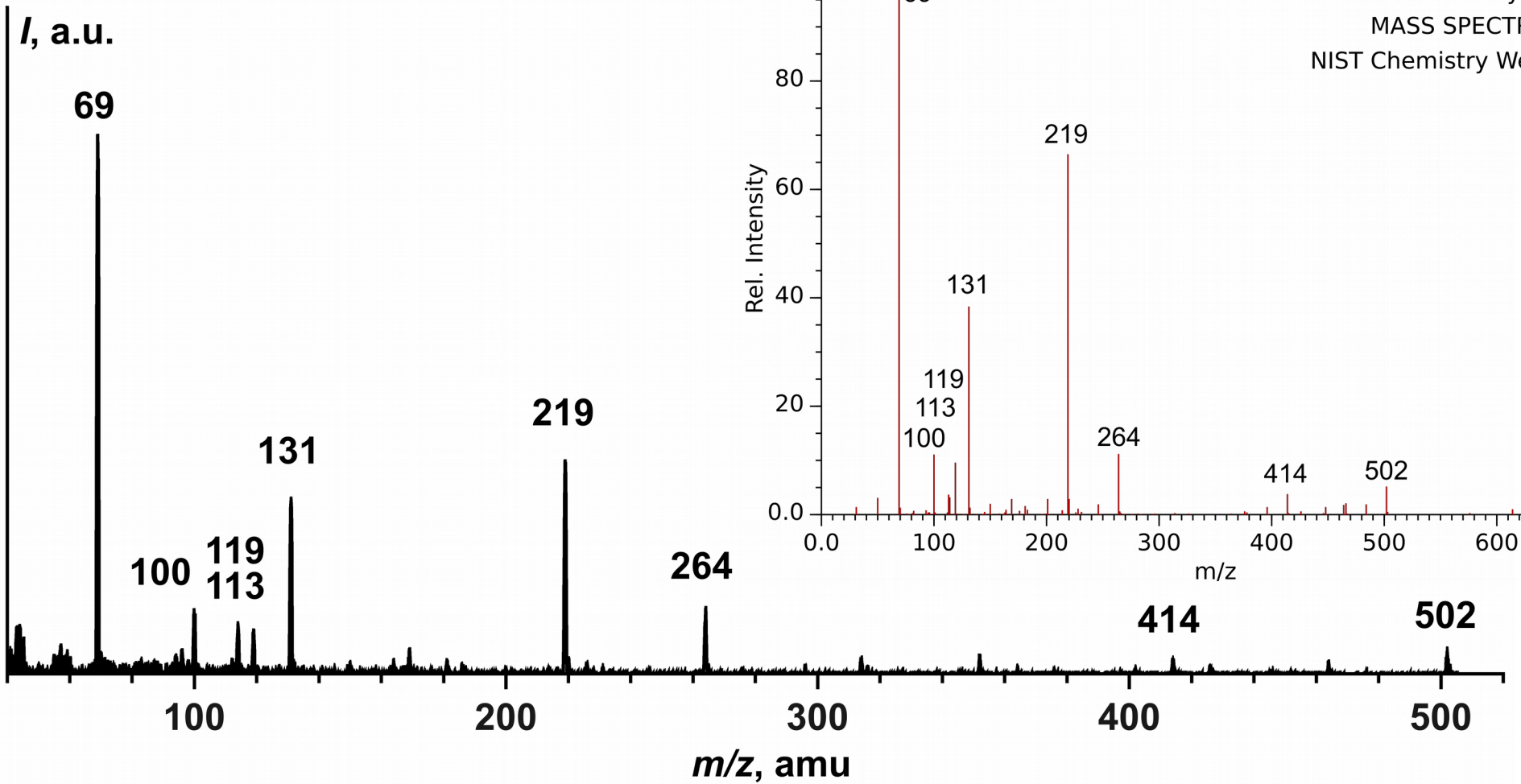


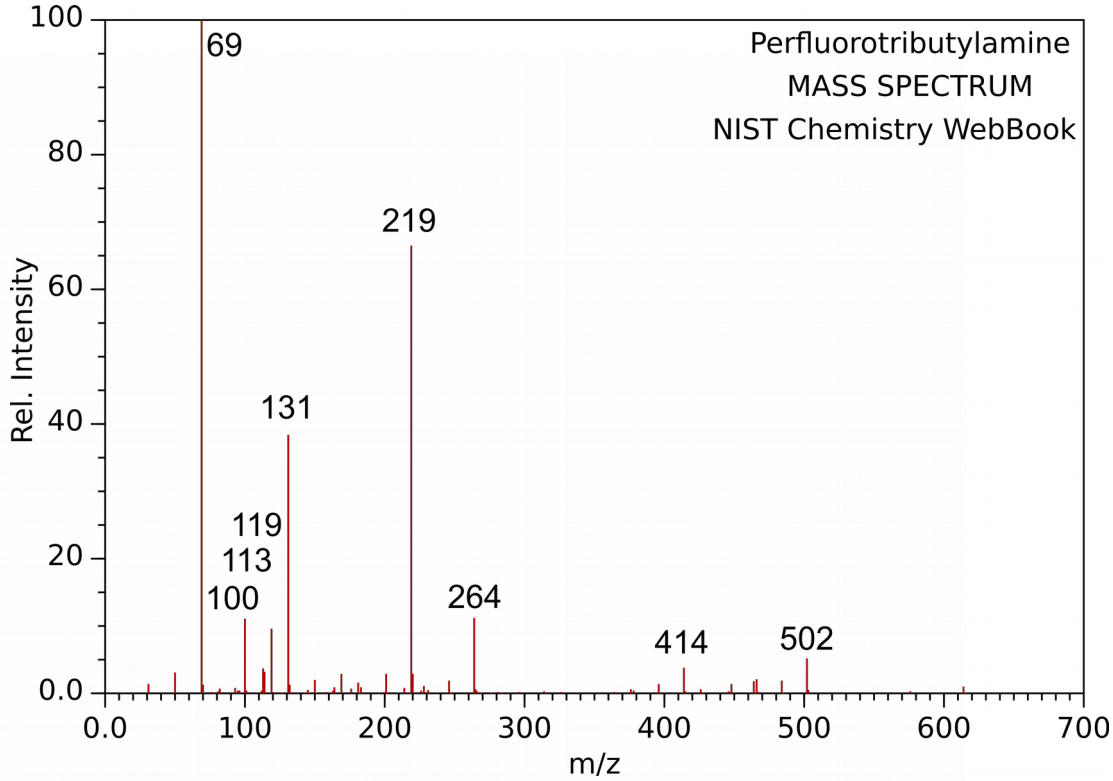
# Calibration




"Heptacosa": Perfluorotributylamine $(C_4F_9)_3N$

# Heptacosa

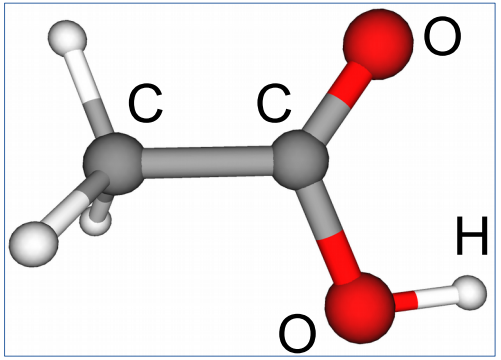

I, a.u.
69
131
219
100
119
113
264
414
502
100
200
300
400
500
m/z, amu


Perfluorotributylamine
MASS SPECTRUM
NIST Chemistry WebBook
Rel. Intensity
m/z
69
219
131
119
113
100
264
414
502
0.0
20
40
60
80
100
0.0
100
200
300
400
500
600
700

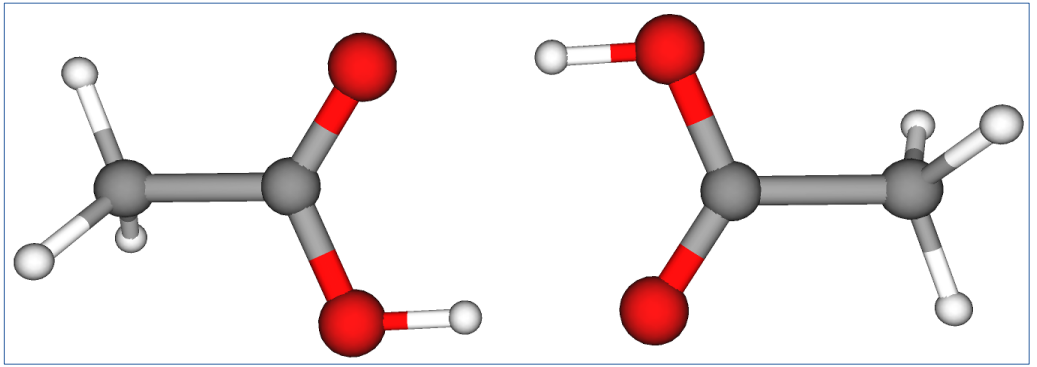
# Acetic acid:
# A combined ED+MS experiment

# Acetic acid

Monomer




Dimer



# History

*JACS*, 66 (1944) 574.

**An Electron Diffraction Investigation of the Monomers and Dimers of Formic, Acetic and Trifluoroacetic Acids and the Dimer of Deuterium Acetate[1]**

By J. Karle[2] and L. O. Brockway

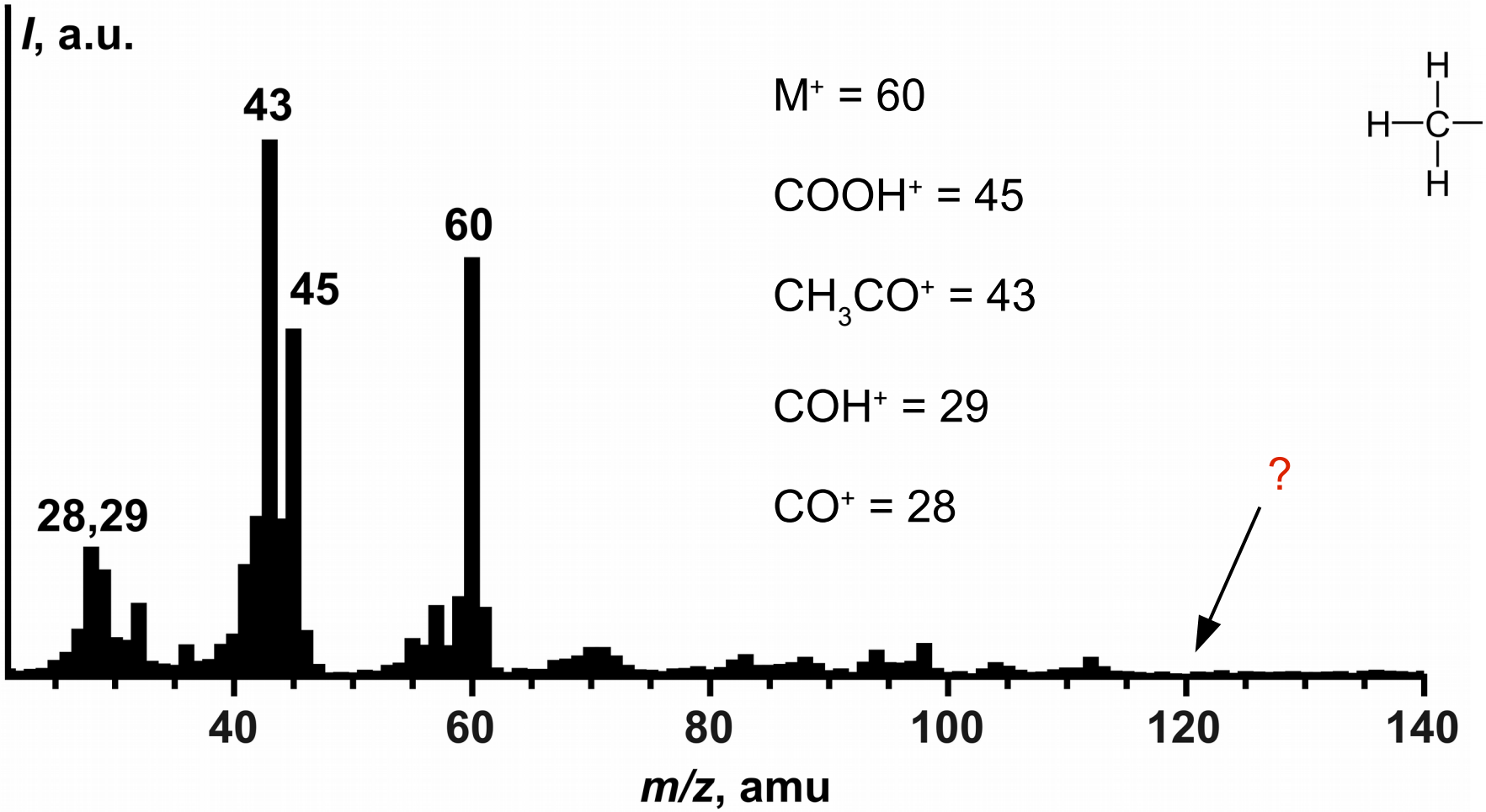
*J. Mol. Struct.*, 7 (1971) 67.

**A REINVESTIGATION OF THE MOLECULAR STRUCTURE OF ACETIC ACID MONOMER AND DIMER BY GAS ELECTRON DIFFRACTION**

J. L. DERISSEN

*Laboratory for Crystal Chemistry, Catharijnesingel 51, Utrecht (The Netherlands)*

# AA mass-spectra @ $T$ = 296 K




$M^+ = 60$

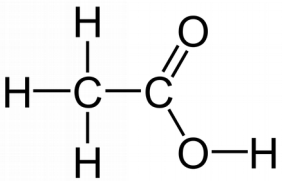
$COOH^+ = 45$

$CH_3CO^+ = 43$

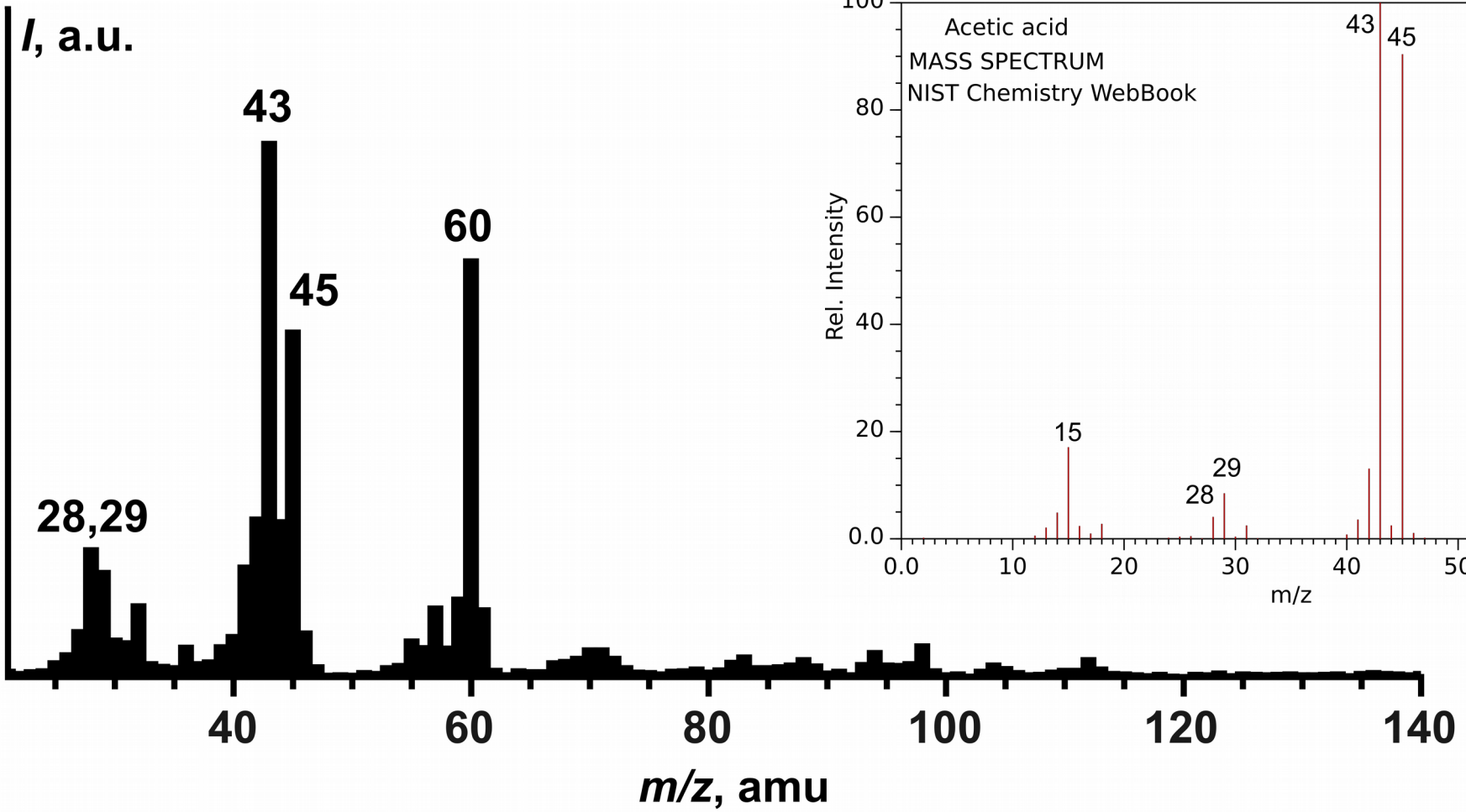
$COH^+ = 29$

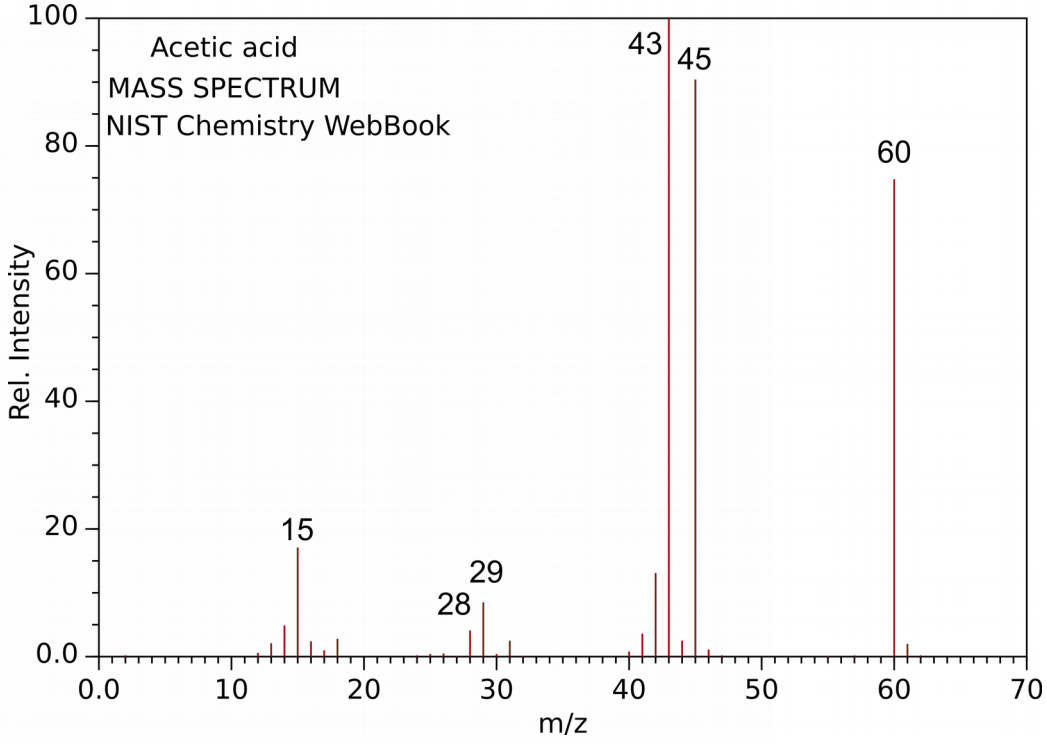
$CO^+ = 28$

?

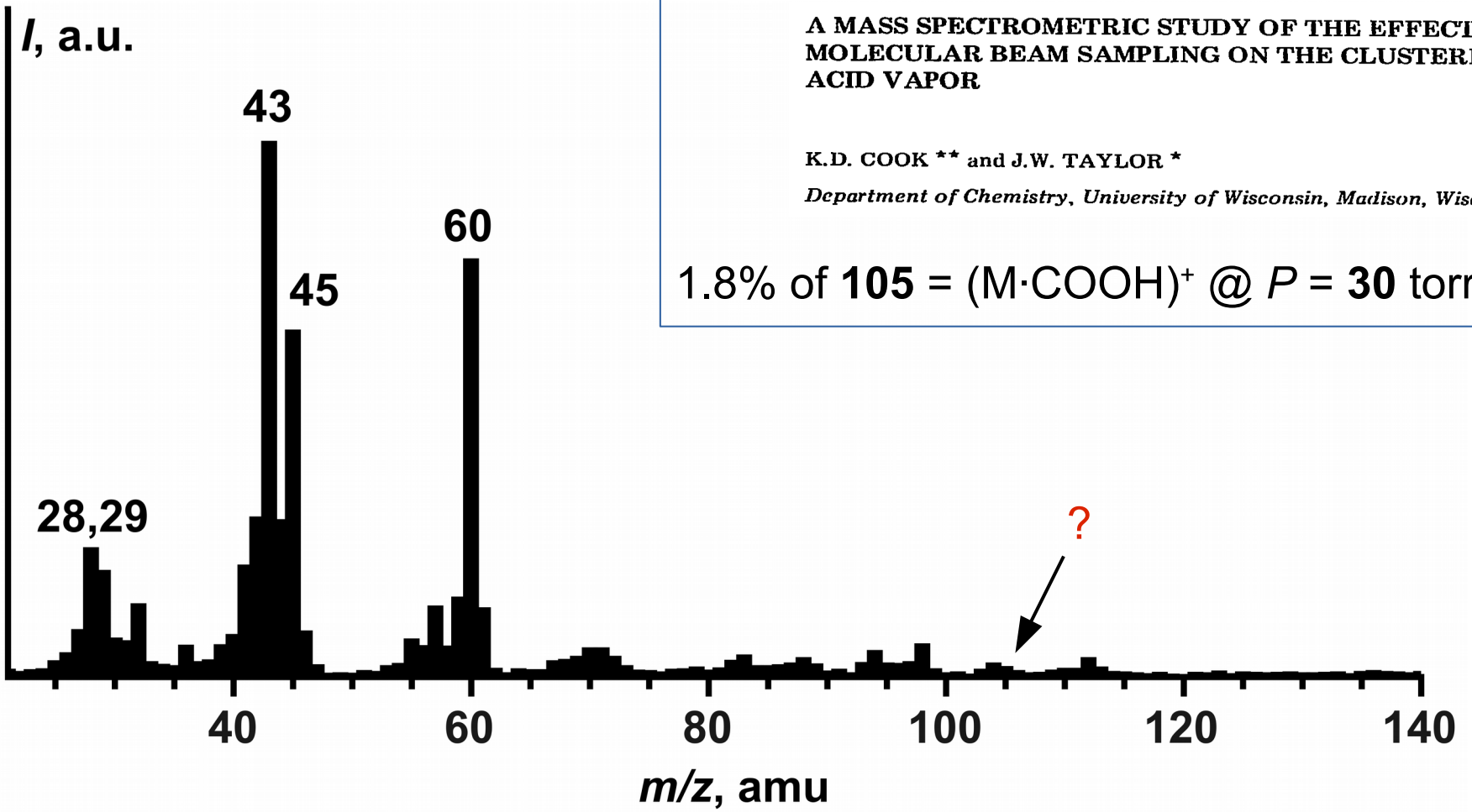


# AA mass-spectra @ $T$ = 296 K



# AA mass-spectra @ $T$ = 296 K



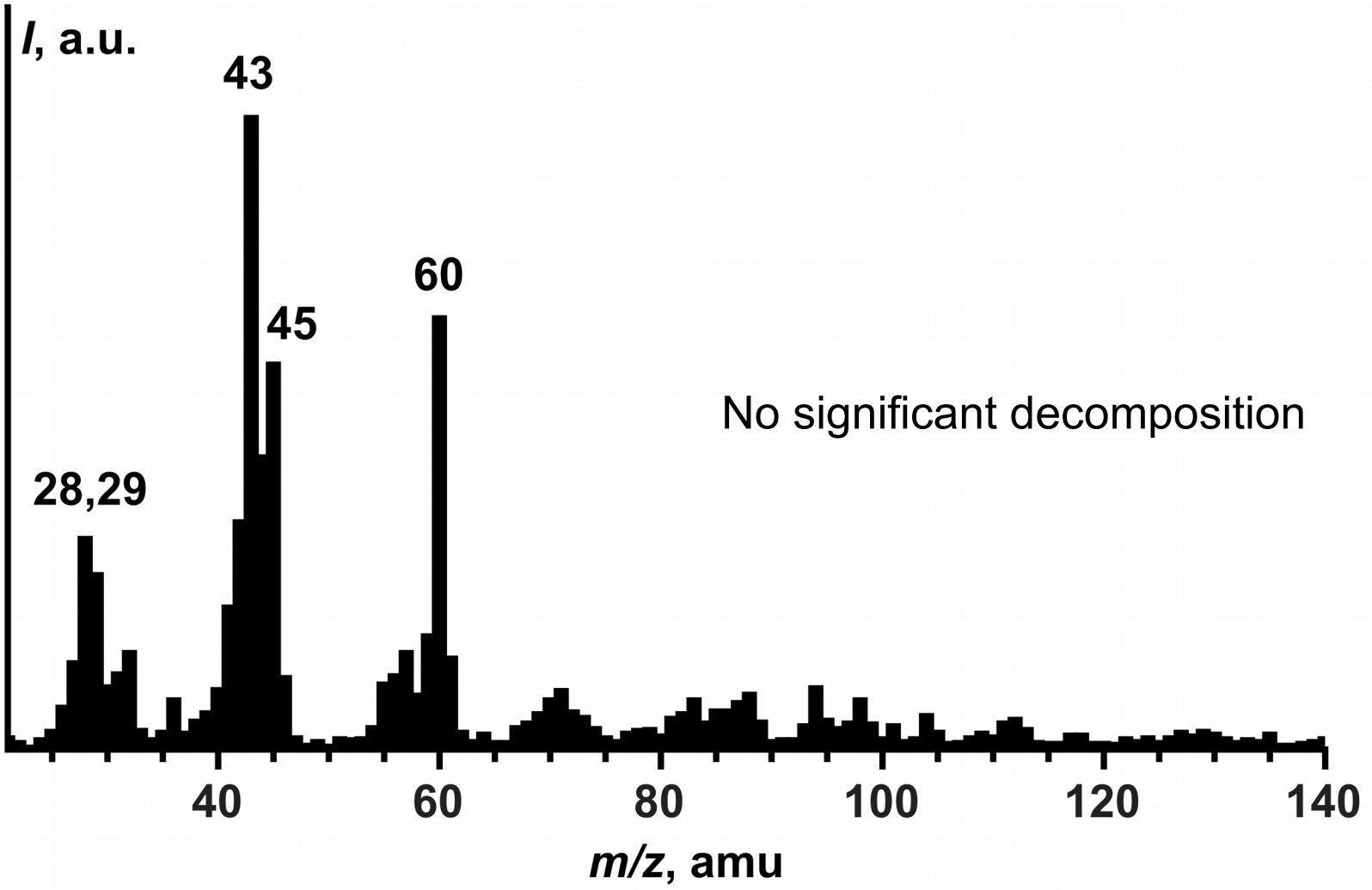

A MASS SPECTROMETRIC STUDY OF THE EFFECT OF SUPERSONIC MOLECULAR BEAM SAMPLING ON THE CLUSTERING OF ACETIC ACID VAPOR

K.D. COOK ** and J.W. TAYLOR *

*Department of Chemistry, University of Wisconsin, Madison, Wisconsin 53706 (U.S.A.)*

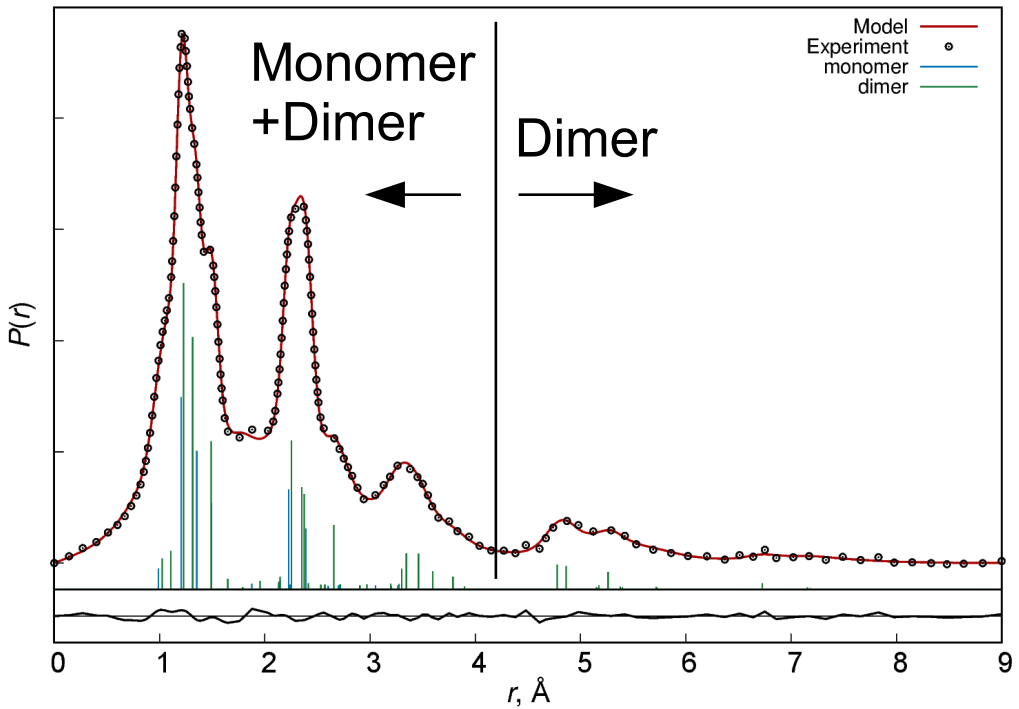
1.8% of **105** = $(M{\cdot}COOH)^+$ @ $P$ = **30** torr, $U$ = 20 eV

# AA mass-spectra @ *T* = 458 K

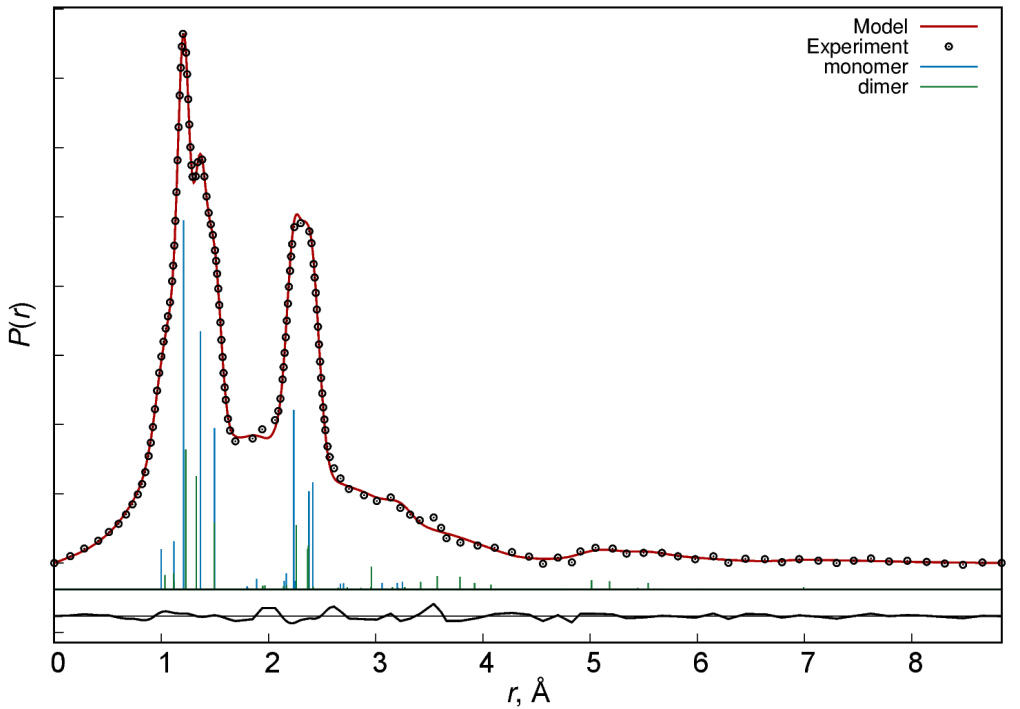


# AA GED

*T* = 296 K
54(3) % monomer




*T* = 458 K
83(5) % monomer



Thank you!