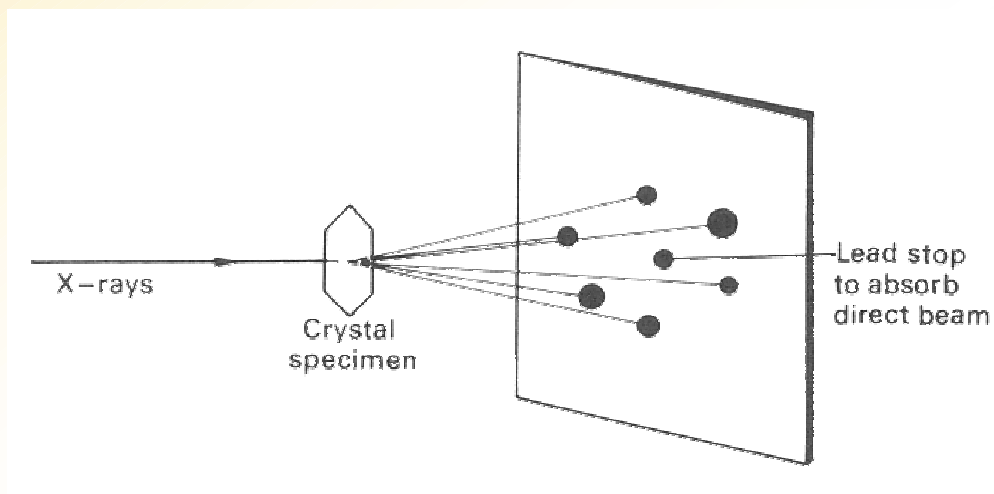


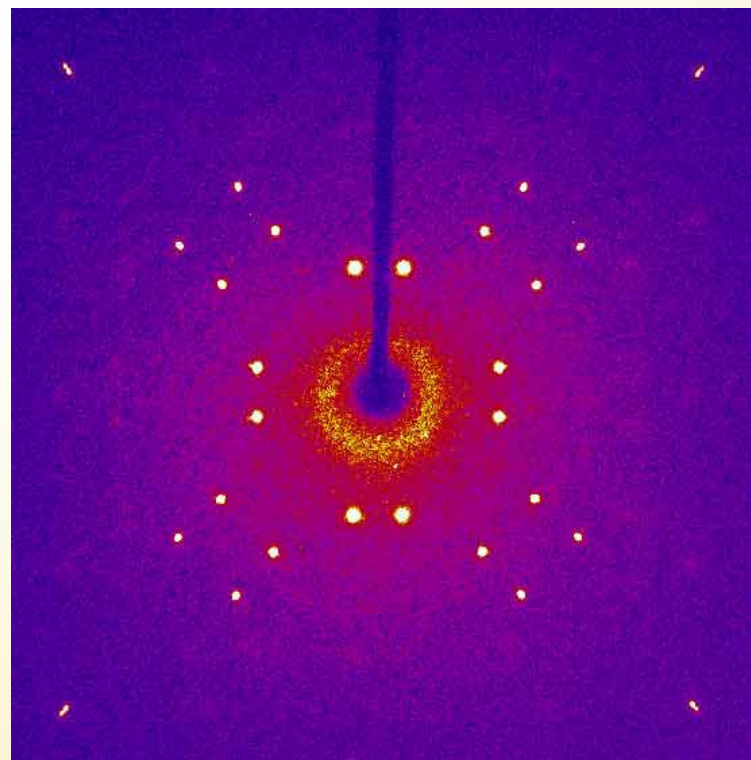
Structure Determination by X-Ray Crystallography using Bruker Nonius X8-APEX

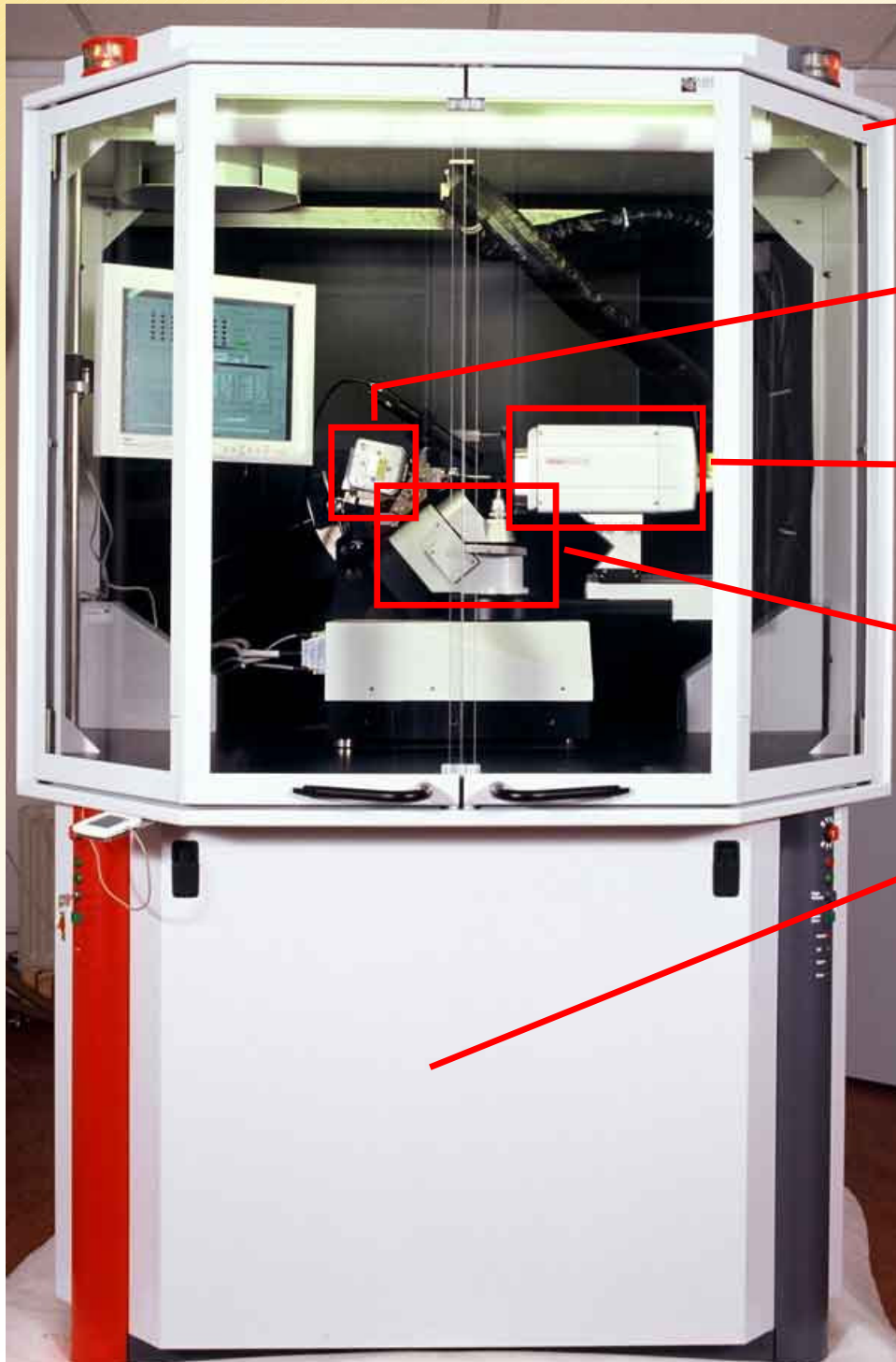
Andrea CORNIA

*Dipartimento di Chimica, Università di Modena e Reggio Emilia & INSTM,
Via G. Campi 183, 41100 MODENA (Italy)*



Single-crystal X-ray diffraction experiment





Cabinet

X-ray tube

SMART APEX detector

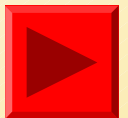
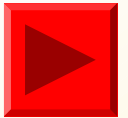
Four-circle
KAPPA goniostat

HT generator + Central controller

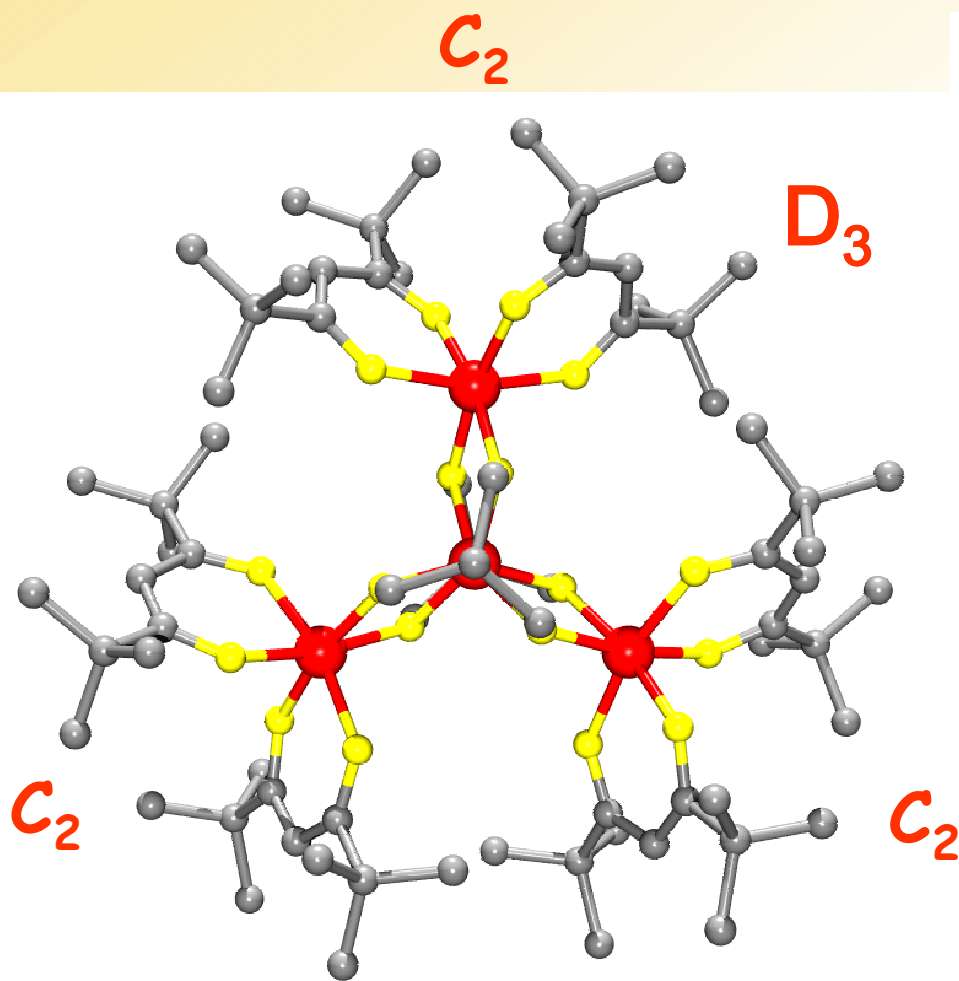
Kryo-Flex Cryostat

Video Microscope

X8 APEX Software



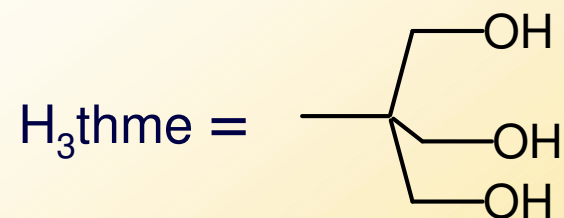
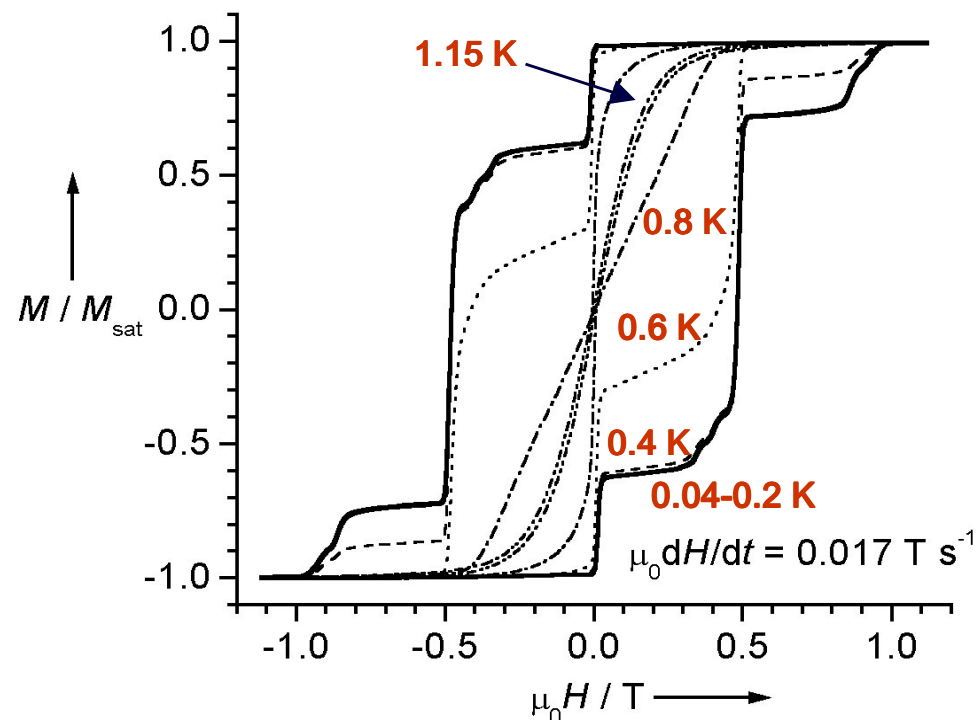
$[\text{Fe}_4(\text{thme})_2(\text{dpm})_6]$: a Tiny Magnet



● Iron(III) ($s = 5/2$)

● Oxygen

● Carbon



$\text{Hdpm} = \text{dipivaloylmethane}$

SiemensP4 RA (in 65 hours)

Experimental Parameters

Empirical formula	$C_{76}H_{132}Fe_4O_{18}$
Formula weight	1557.22
Crystal size	$0.28 \times 0.23 \times 0.21$ mm
Temperature	203(2) K
Wavelength	0.71073 Å

Unit Cell Determination

Crystal system	Trigonal
Space group	$R\bar{3}c$
Unit cell dimensions	
$a = 16.353(1)$ Å	$\alpha = 90^\circ$
$b = 16.353(1)$ Å	$\beta = 90^\circ$
$c = 56.917(6)$ Å	$\gamma = 120^\circ$
Volume	13181.6(18) Å³
Z	6

Data Collection

Time required	65^h
Scan type	θ - 2θ
Theta range	2.50 to 25.0°
Reflections collected	5701
Independent reflections	2584 [$R(\text{int}) = 0.0456$]
Redundancy	2.2
Average $I/\sigma(I)$	17.73

Structure Refinement

Data / restraints / parameters	2584 / 7 / 172
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0454$, $wR2 = 0.1080$
R indices (all data)	$R1 = 0.0799$, $wR2 = 0.1234$
Largest diff. peak and hole	0.251 and -0.262 eÅ ⁻³

Bruker-Nonius X8 APEX (in 11 hours)

Experimental Parameters

Empirical formula	$C_{76}H_{132}Fe_4O_{18}$
Formula weight	1557.22
Crystal size	$0.28 \times 0.23 \times 0.21$ mm
Temperature	120(2) K
Wavelength	0.71073 Å

Unit Cell Determination

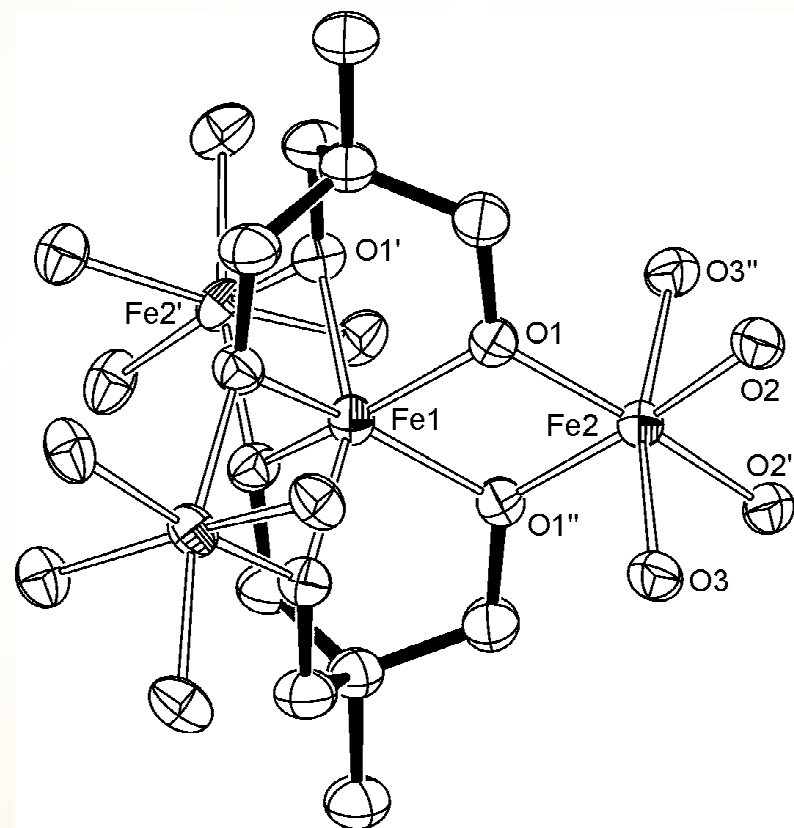
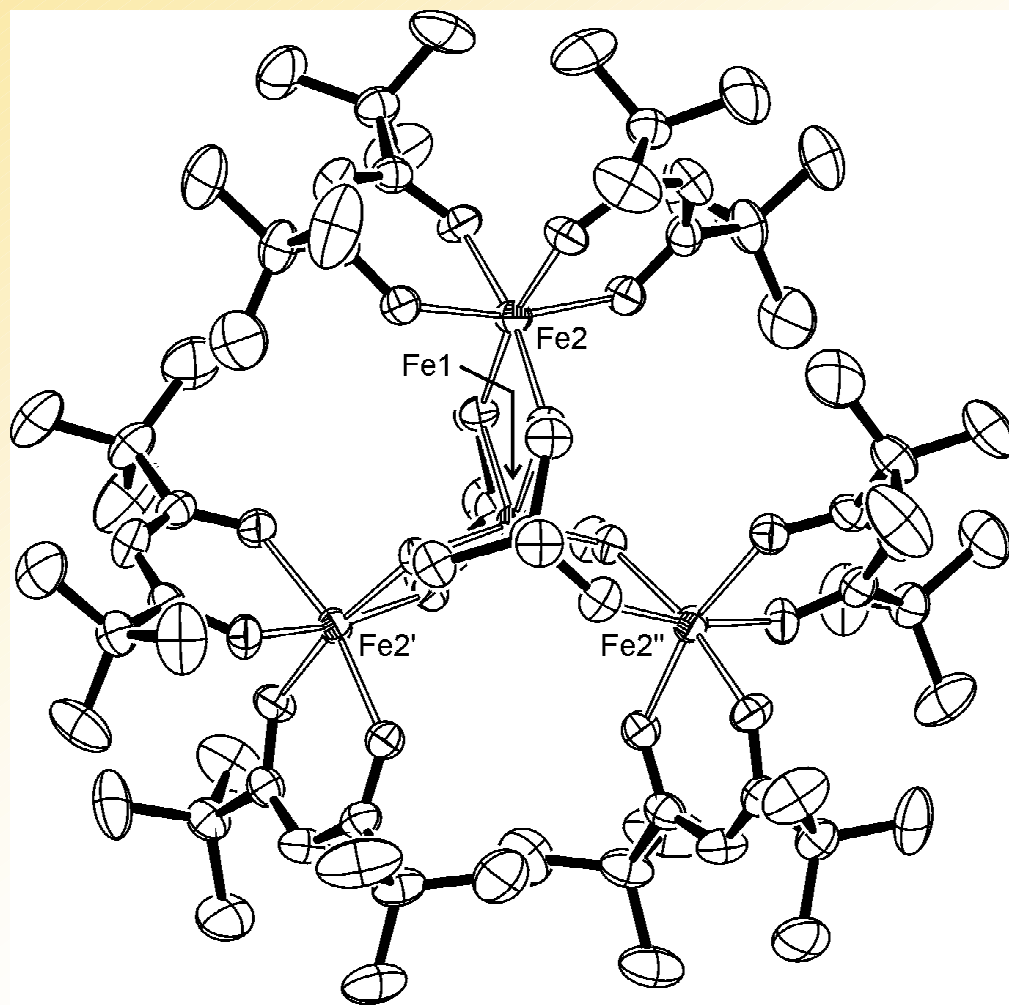
Number of frames	60 (3 runs)
Exposure time	5''
Time required	11'
Scan type	ω , ω , ω
Scan range	0.3°
Detector-to-sample distance	40 mm
Crystal system	Trigonal
Space group	$R\bar{3}c$
Unit cell dimensions	
$a = 16.2069(2)$ Å	$\alpha = 90^\circ$
$b = 16.2069(2)$ Å	$\beta = 90^\circ$
$c = 57.1099(8)$ Å	$\gamma = 120^\circ$
Volume	12991.0(3) Å³
Z	6

Data Collection

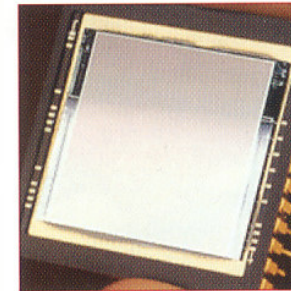
Number of frames	1999 (3 runs)
Exposure time	10''
Time required	10^h 20'
Scan type	ϕ , ϕ , ϕ
Scan range	0.3°
Detector-to-sample distance	40 mm
Theta range	2.99 to 28.27°
Reflections collected	44659
Independent reflections	3571 [$R(\text{int}) = 0.0415$]
Redundancy	12.5
Average $I/\sigma(I)$	34.60
Completeness to $\theta = 28.27^\circ$	99.4 %

Structure Refinement

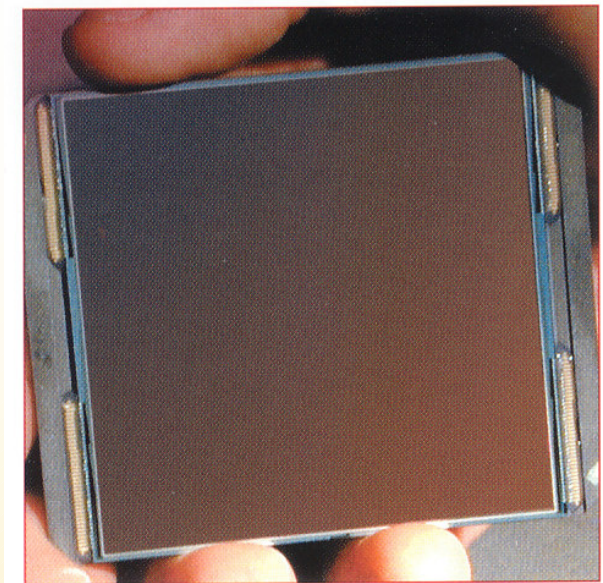
Data / restraints / parameters	3571 / 7 / 172
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0381$, $wR2 = 0.1022$
R indices (all data)	$R1 = 0.0606$, $wR2 = 0.1131$
Largest diff. peak and hole	0.657 and -0.198 eÅ ⁻³



SMART APEX detector

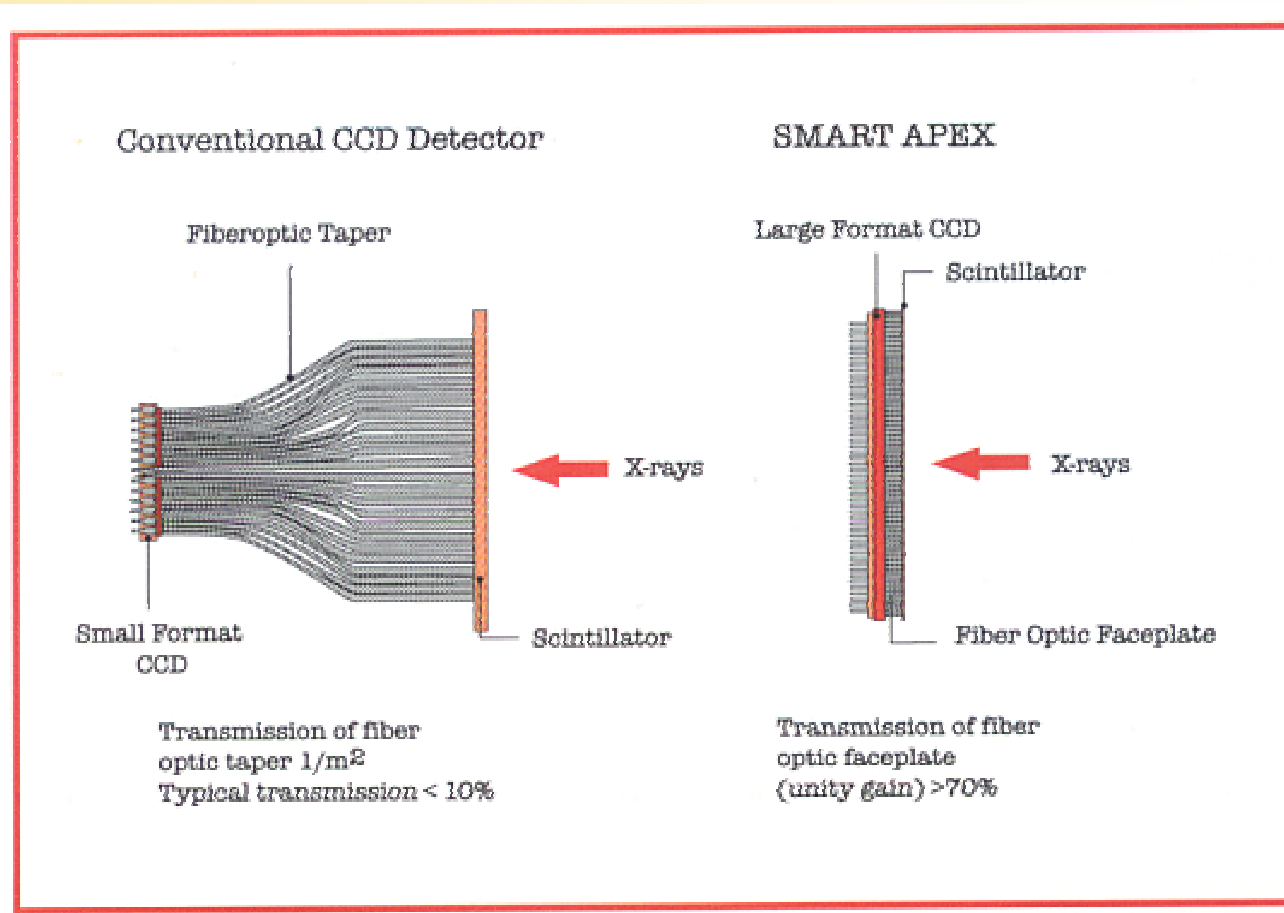


*Comparative size of
older 1K CCD chip
(left) and 4K CCD
chip (right)*



62 × 62 mm CCD Chip, 4096 × 4096 pixel, 1 pixel = 15 μ m

SMART-APEX detector

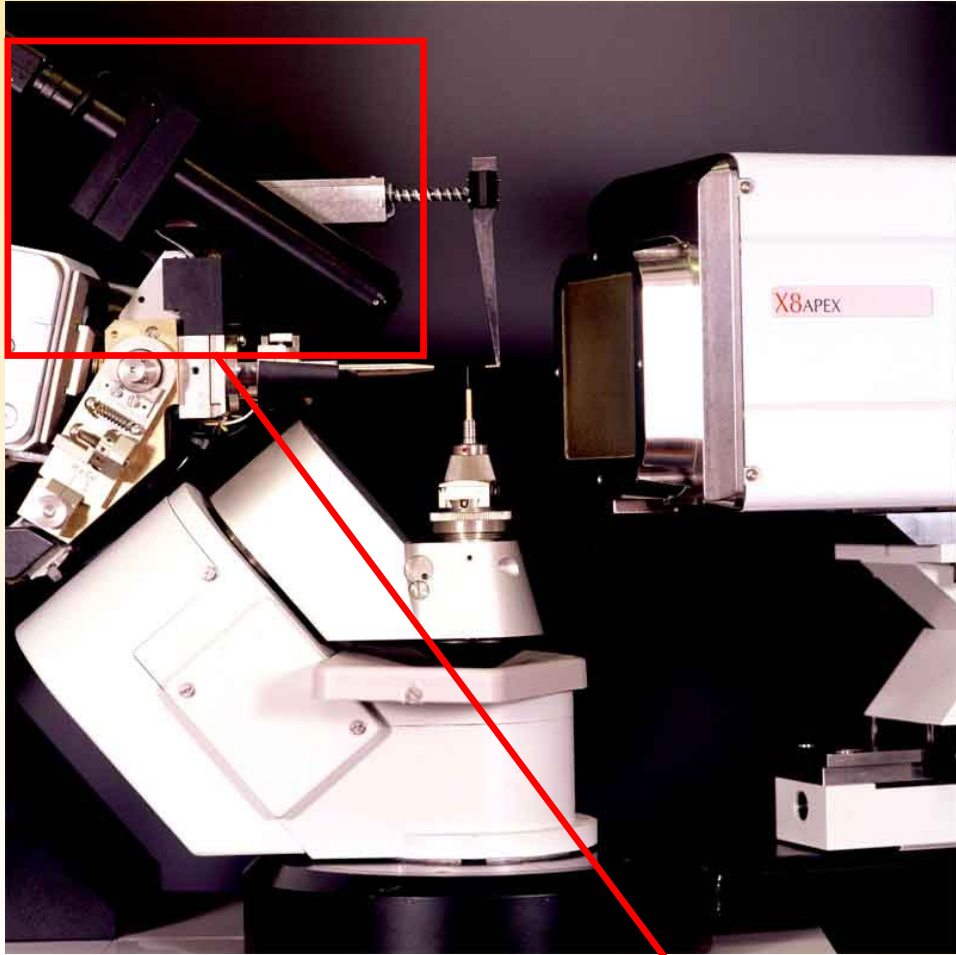


No fiberoptic taper between CCD and scintillator screen

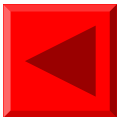
- 1:1 imaging
- extremely high sensitivity (~170 electrons per Mo X-ray photon)
- no spatial distortion from the fiber



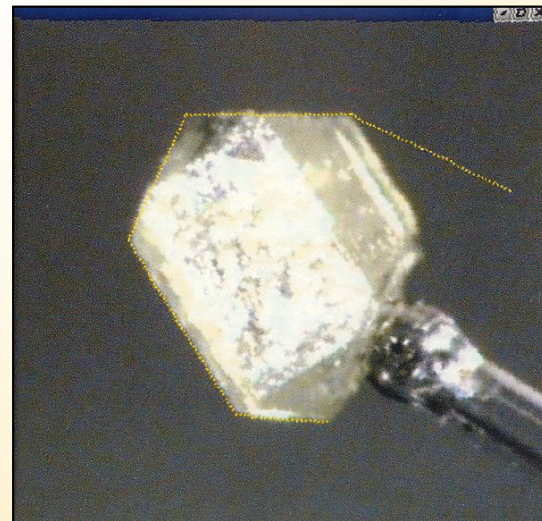
Four-circle KAPPA goniostat



- “open” geometry, minimum obstruction
 - full sample-positioning freedom
 - ultimate flexibility for oriented exposures
 - sample conditioning devices
 - high resolution work ($-90^\circ < 2\theta < 135^\circ$)
- short beam paths
- $\leq 5\text{-}\mu\text{m}$ sphere of confusion
- computerized detector distance (35–165 mm)



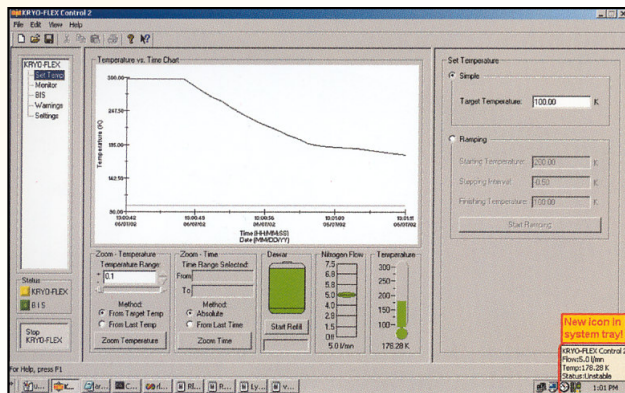
Video Microscope
40 - 120 x



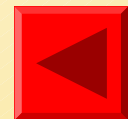
Kryo-Flex low-temperature attachment



- $T = 90 - 300 \text{ K } (\pm 0.1 \text{ K})$
- cold N_2 gas stream (from liq. N_2 , $< 0.5 \text{ l/h}$)
- automatic refill of 60-l dewar from external tank
- dry-air shield to avoid ice formation
- digital T controller and graphical software interface

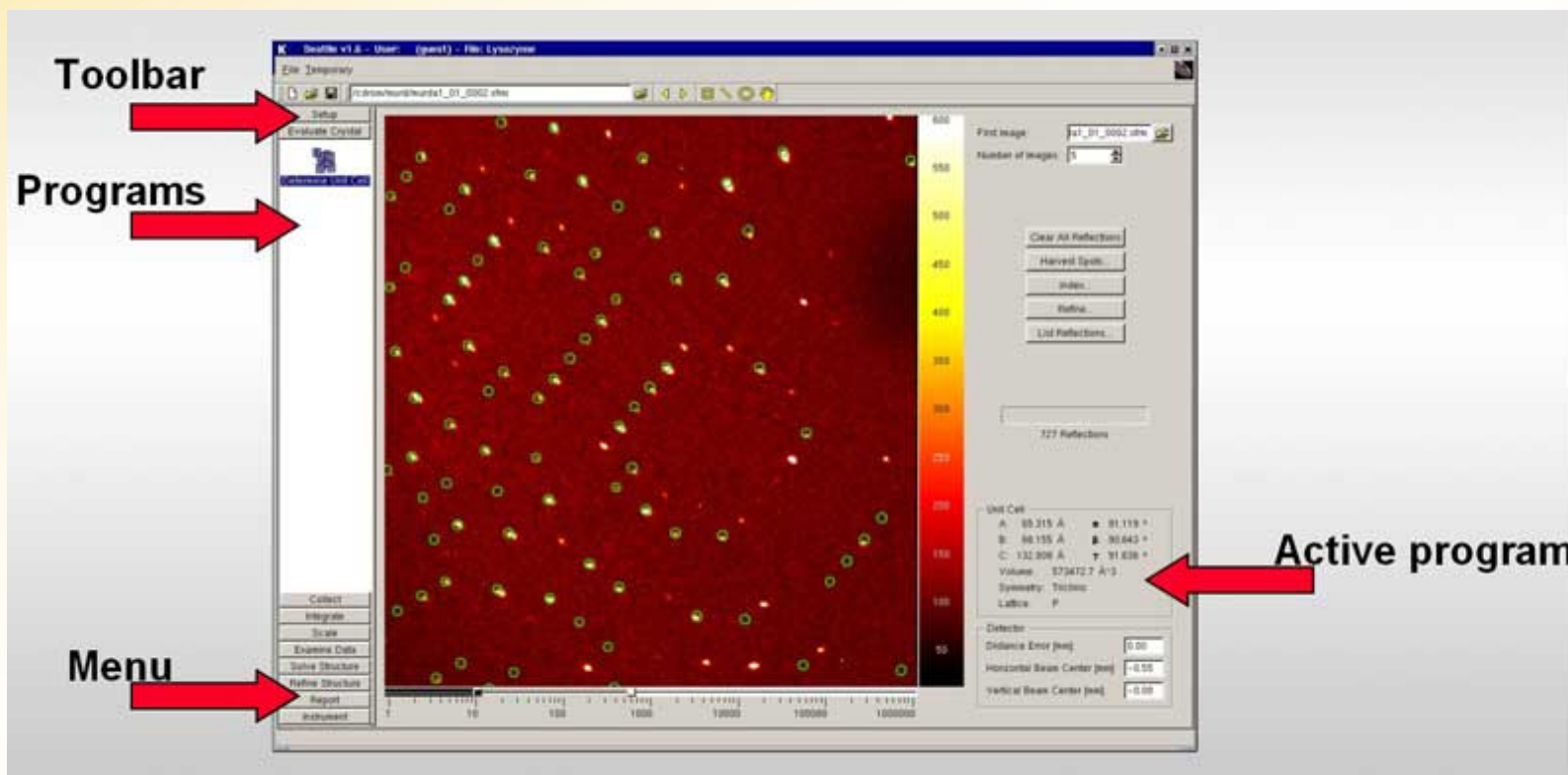


- reduced thermal motions, superior data quality
- simpler handling of air-sensitive crystals
- on-site crystal mounting



X8 APEX Software

Indexing → Data collection → Integration → Scaling & Absorption Correction
→ Space group assignment → Structure solution and refinement
→ Graphics & Publication Material



Automatic optimization of data collection parameters (strategy)
Reciprocal lattice plots
Separate integration of crystal domains for twinned samples

