Overfocused-probe ptychography of a 25–45 nm thick sample

Spectra Ultra, 300 kV, EMPAD (128 × 128 px). Experiment-design note, 2026-10-02, version 4: built on the literature setting given by the facility (21 nm sample) and the 300 kV camera-length calibration in this folder (data 2026-08-21). Figures 1–3 and 6 use a nominal 40 nm; the thickness table covers 25–45 nm.

Recommendation

Setting	Value	Why
Sample thickness	measure first	the overfocus depends on it (EELS log-ratio t/λ on the GIF)
Accelerating voltage	300 kV	as calibrated
Probe semi-convergence α	21.5 mrad	as in the literature (21.4 mrad)
EMPAD camera length	430 mm	window 2.93 nm; detector edge 2.0α (literature 2.5α)
Overfocus C1 (CEOS sign)	≈ 50 nm − t	+10 nm at 40 nm; keeps the literature's 0.73 margin (thickness table below)
Scan step	0.41 Å (0.4–0.5 Å)	90 % overlap (literature 95 %)
Probe current	33 pA (as literature)	adjust to the sample's dose tolerance
Expected BF disc radius	≈ 32 px	check on the EMPAD before collecting (Figure 5)

- Probe: 0.43 nm at the entrance surface, 2.15 nm at the exit, inside the 2.93 nm window with margin (Figure 6).
- Scan as in the literature: 128 × 128 positions at 0.41 Å = 5.2 nm field of view (about 21 Mx, scaling the 1.8 Mx = 2.38 Å / 256 px setting in the calibration notes).
- Dose at 33 pA and 0.41 Å: 1.2e+06 e/Å² for each millisecond of dwell per position. The dwell (EMPAD frame time) was not given; at 1 ms a 128 × 128 scan takes about 16 s.
- Why +10 nm: it puts the exit probe at 0.73 of the window, the same margin as the literature setting on its 21 nm sample (0.73), which is known to reconstruct well.
- Bolder: 430 mm with C1 = +15 nm gives 94 % overlap but 0.81 of the window, beyond the proven margin.
- To keep the literature's 20 nm overfocus: 550 mm with C1 = +20 nm (exit probe 0.70 of the window, 95 % overlap), at less detector range (edge 1.6α instead of 2.0α).
- If the thickness can't be measured: 550 mm with C1 = +15 nm works across 25–45 nm (exit probe 0.47–0.70 of the window, 94 % overlap at 0.41 Å).
- Without overfocus: focus at mid-depth (about t/2 into the sample); the probe stays ≤ 1 nm through the whole thickness and the literature's camera length (~350 mm) becomes usable.

Your sample: 25–45 nm thick

The overfocus that keeps the literature's margin (exit probe = 0.73 of the window) is C1 = 0.73 × W / (2α) − t: the thicker the sample, the less overfocus. At 21.5 mrad that is about 50 nm − t at 430 mm and 63 nm − t at 550 mm.

Thickness t	430 mm: C1 / overlap	550 mm: C1 / overlap	550 mm, +15 nm: exit/W / overlap
25 nm	+25 nm / 96 %	+38 nm / 97 %	0.47 / 94 %
30 nm	+20 nm / 95 %	+33 nm / 97 %	0.53 / 94 %
35 nm	+15 nm / 94 %	+28 nm / 97 %	0.58 / 94 %
40 nm	**+10 nm / 90 %**	**+23 nm / 96 %**	**0.64 / 94 %**
45 nm	+5 nm / 81 %	+18 nm / 95 %	0.70 / 94 %

- Overlap is at the 0.41 Å step. At 430 mm a 45 nm sample leaves only +5 nm of overfocus, and the overlap drops to about 81 %; above about 40 nm, 550 mm is the better camera length.
- Measure the thickness on the area you will scan (EELS log-ratio t/λ; the inelastic mean free path at 300 kV is of order 100–150 nm, depending on the material), then read C1 off this table.
- Unknown thickness: 550 mm with +15 nm stays at or under the literature's margin over the whole range (dashed line in Figure 7).

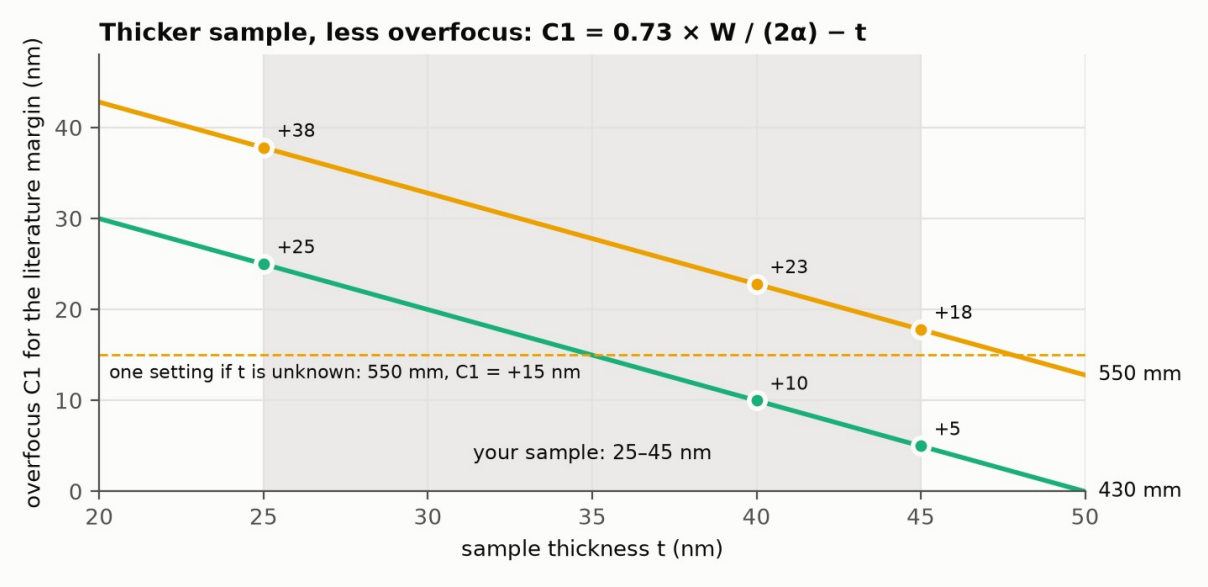


Figure 7. Overfocus that keeps the literature's 0.73 margin vs sample thickness, at 430 and 550 mm. The shaded band is your 25–45 nm; the dashed line is the single setting for an unknown thickness.

The literature setting, and why it changes for a thicker sample

Literature setting (given by the facility): 21.4 mrad, 33 pA, 0.041 nm step over a ≈ 5 nm area, EMPAD 128 px at 0.823 mrad/px, 20 nm overfocus. Taken as 300 kV. With this microscope's calibration:

	Literature	Recommended (430 mm)	Alternative (550 mm)
α	21.4 mrad	21.5 mrad	21.5 mrad
Angle per pixel	0.823 mrad/px	0.672 mrad/px	0.535 mrad/px
Camera length here	≈ 351 mm	430 mm	550 mm
BF disc radius / of width	26 px / 0.41	32 px / 0.50	40 px / 0.63

Detector edge	52.7 mrad = 2.5α	43.0 mrad = 2.0α	34.2 mrad = 1.6α
Window W	2.39 nm	2.93 nm	3.68 nm
Overfocus C1	+20 nm	+10 nm	+20 nm
Exit probe / window, 40 nm	1.07 (too big)	0.73	0.70
Scan step / overlap	0.41 Å / 95 %	0.41 Å / 90 %	0.41 Å / 95 %

- The literature's 0.823 mrad/px is about 351 mm on this microscope (between 340 and 430 mm), and its bright-field disc spans 0.41 of the detector width.
- At 20 nm overfocus the probe at the exit of the literature's 21 nm sample is 1.75 nm, 0.73 of that window: a margin known to reconstruct well, and the benchmark used here. For a 40 nm sample it is 2.6 nm, 1.07 of the window: it does not.
- So a 40 nm sample has to give something up: detector range (a longer camera length) or overfocus (a smaller C1). 430 mm with +10 nm keeps the detector edge closest to the literature's (2.0α vs 2.5α) and matches its 0.73 margin on the window; Figure 6 compares the options.
- The literature's 20 nm is overfocus; this note uses the CEOS convention, C1 > 0 = overfocus. Confirm on this column with the nudge test (checklist, item 1).

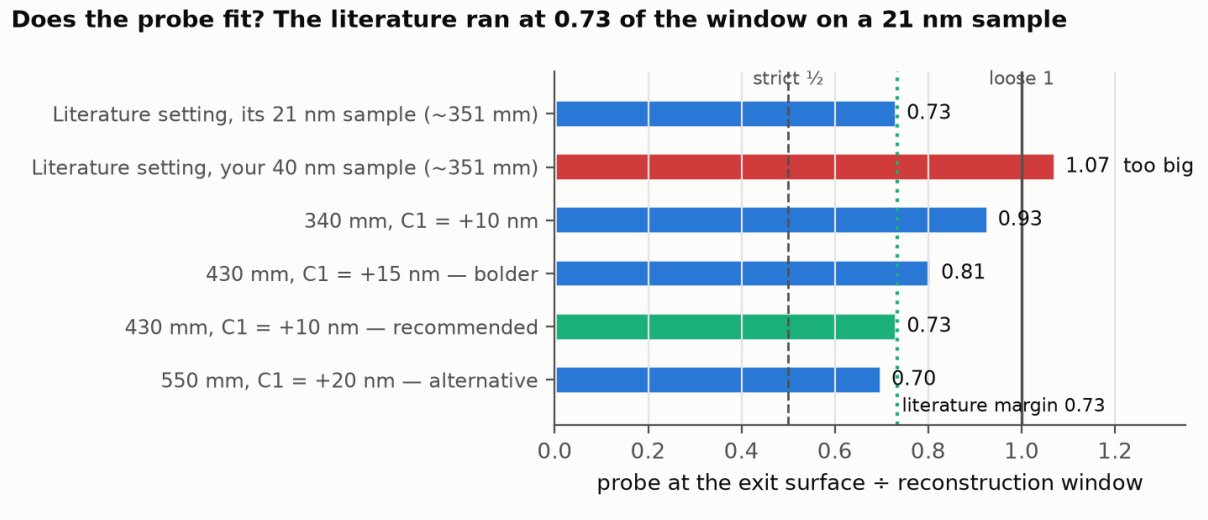


Figure 6. The exit-surface probe as a fraction of the reconstruction window, for the literature setting and for the options on this microscope (40 nm sample unless stated).

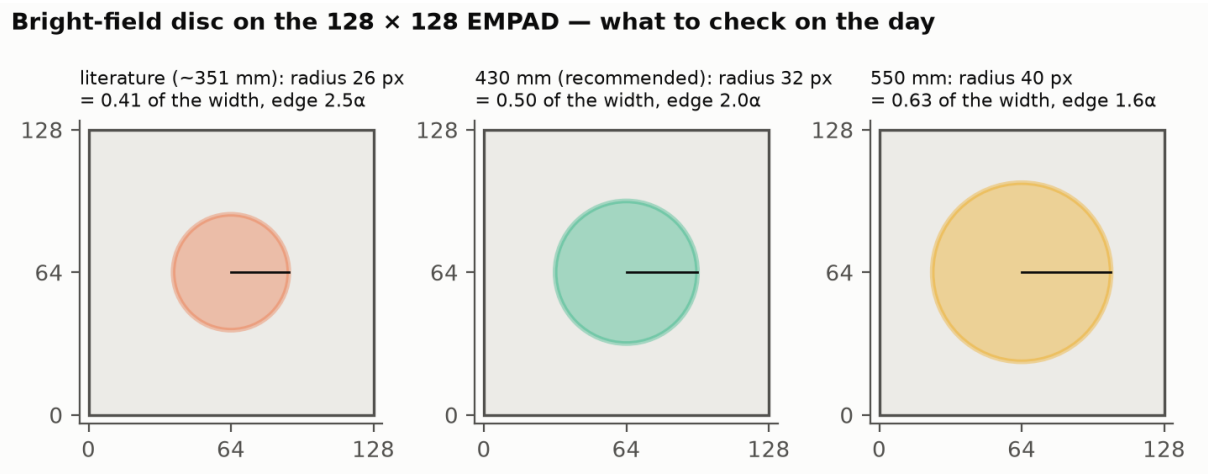


Figure 5. The bright-field disc on the 128 × 128 EMPAD at the literature setting, the recommended 430 mm and the 550 mm alternative.

1. Why the sample thickness limits the overfocus

With overfocus the probe crossover sits above the entrance surface, so the probe keeps widening on its way through the sample. Its geometric diameter at depth z below the entrance surface is D(z) = 2α(C1 + z); at the exit surface of a 40 nm sample it is 2α(C1 + 40 nm). The reconstruction can only hold a probe that fits inside its real-space window W = λ / Δθ, where Δθ is the EMPAD's angle per pixel. A probe larger than the window wraps around and the reconstruction fails or converges poorly.

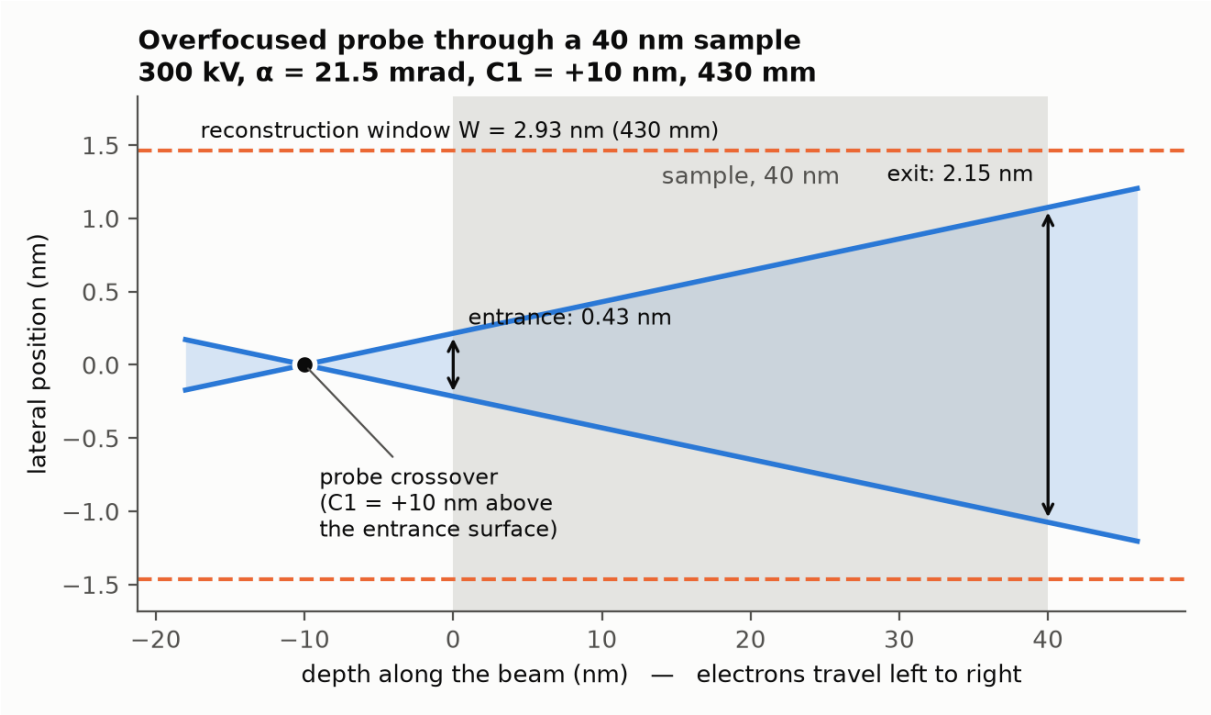


Figure 1. The recommended geometry to scale: C1 = +10 nm at 430 mm. The probe grows from 0.43 nm at the entrance to 2.15 nm at the exit, inside the 2.93 nm window.

2. How much overfocus fits

Two rules of thumb bound the largest usable C1: a strict one (exit probe ≤ half the window, safe against probe tails) and a loose one (exit probe ≤ the window). Solving 2α(C1 + t) ≤ f·W gives C1_max = f·W / (2α) − t, with f = ½ or 1. The literature setting ran at f = 0.73 on its 21 nm sample, which is the margin this note aims for.

Camera length	mrad/px	Window W	Detector edge	BF radius	C1 max strict	C1 max loose
270 mm	1.058	1.86 nm	68 mrad (3.1 α)	20 px	—	+3 nm
340 mm	0.851	2.31 nm	54 mrad (2.5 α)	25 px	—	+14 nm
430 mm	**0.672**	**2.93 nm**	**43 mrad (2.0 α)**	**32 px**	**—**	**+28 nm**
550 mm	0.535	3.68 nm	34 mrad (1.6 α)	40 px	+3 nm	+46 nm

At 21.5 mrad, 430 mm allows up to +28 nm (loose), so +10 nm leaves margin; 340 mm allows only +14 nm and 270 mm almost nothing for a 40 nm sample.

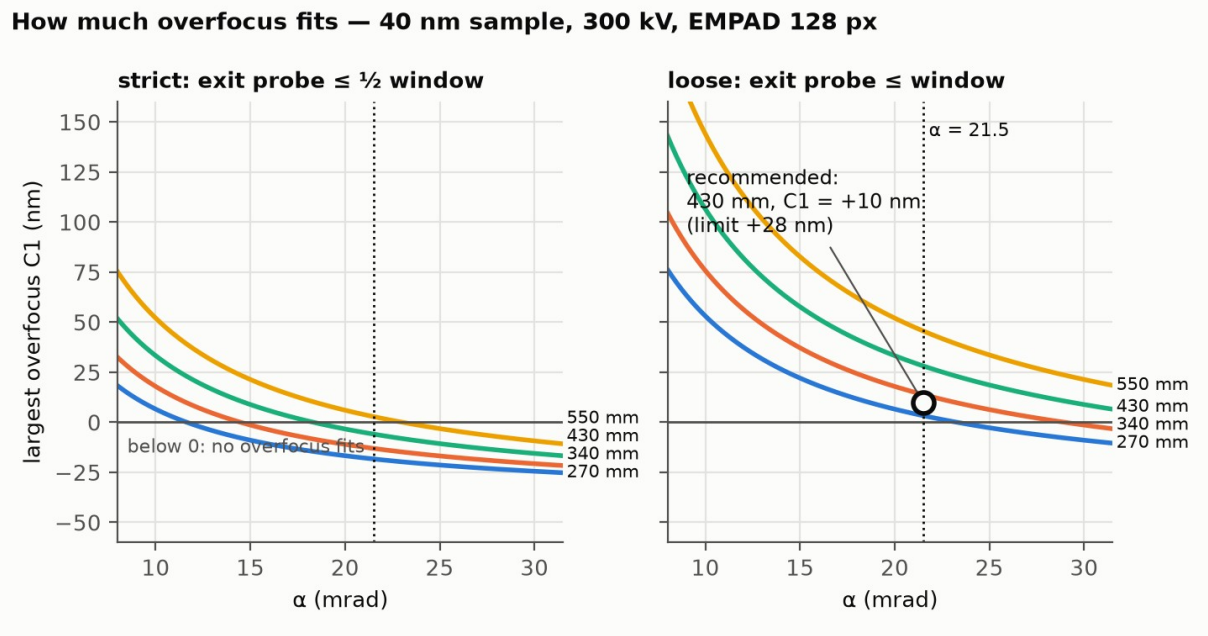


Figure 2. Largest overfocus vs convergence angle for each camera length; left the strict limit, right the loose limit. Below zero no overfocus fits. The ring marks the recommendation (C1 = +10 nm at 430 mm, against that camera length's +28 nm limit).

3. Scan step and overlap

Neighbouring probe positions must overlap. 70 % is the minimum usually quoted; the literature setting uses 95 %. With overfocus the probe is smallest at the entrance surface, so that is where the overlap 1 − step / D(entrance) is checked. It depends on α, C1 and the step, not on the camera length.

C1	Probe entrance → exit	0.41 Å	0.5 Å	1.0 Å	2.38 Å
+10 nm	**0.43 → 2.15 nm**	**90 %**	**88 %**	**77 %**	**45 %**
+15 nm	0.64 → 2.37 nm	94 %	92 %	84 %	63 %
+20 nm	0.86 → 2.58 nm	95 %	94 %	88 %	72 %

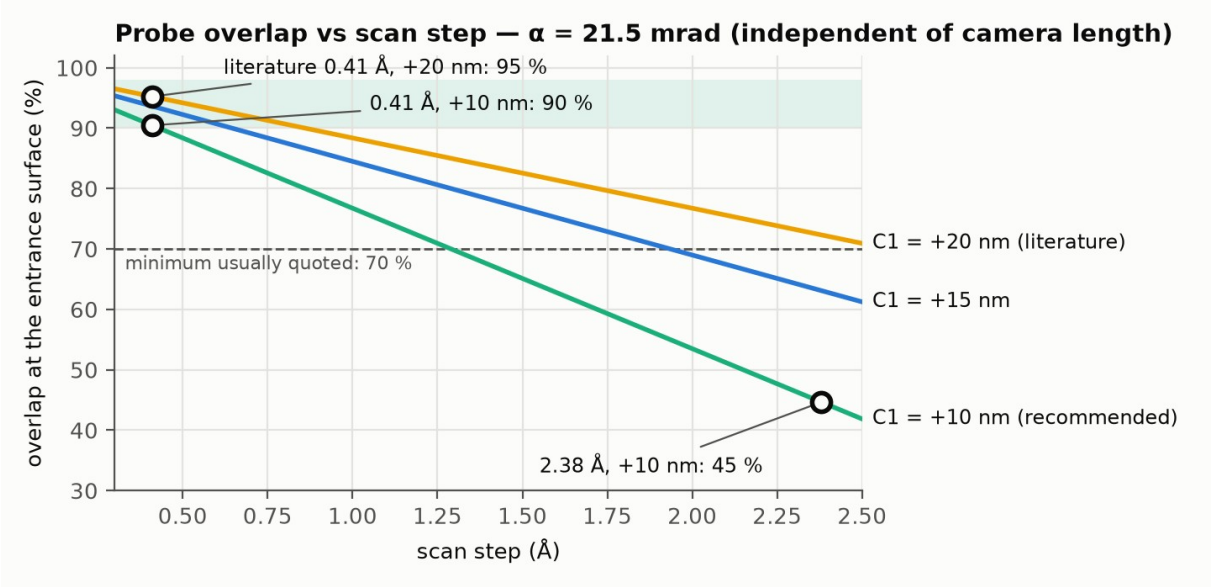


Figure 3. Overlap vs scan step at α = 21.5 mrad; shaded: dense overlap, 90–98 %; dashed: the 70 % minimum. The recommended C1 = +10 nm at the literature's 0.41 Å gives 90 % (the literature's +20 nm: 95 %); a 2.38 Å step would give only 45 %.

4. Before collecting — checklist

1. Measure the sample thickness on the scan area (EELS log-ratio) and take C1 from the thickness table.
2. Confirm the C1 sign once on this column: note the TEM UI Defocus, apply a small known C1 (e.g. +20 nm) in DCORprime, read Defocus again. If it rose by about 20 nm, C1 > 0 is overfocus (CEOS convention). If it fell, the sign is reversed here and overfocus needs C1 < 0.
3. Check the bright-field disc on the EMPAD at 430 mm: radius about 32 px, diameter about 0.50 of the width (Figure 5). A different size means α or the camera length differs from the calibration, and the numbers above shift.
4. Set the reconstruction's starting probe to C1 = +10 nm with the matching sign convention (PtyRAD and AutoACEM's twin: overfocus positive; abTEM: defocus = −C1).
5. Set the scan to 128 × 128 at 0.41 Å (≈ 21 Mx) and choose the dwell and current for the dose the sample tolerates.

5. Caveats

- The limits come from rules of thumb (exit probe ≤ ½ or 1 × window, geometric probe size), not from measured reconstruction failures. Diffraction broadening (≈ λ/α ≈ 0.09 nm) is ignored.
- The literature's voltage and dwell were not given; 300 kV is assumed, and the dose is quoted per millisecond of dwell. Its sample was 21 nm thick.
- Your sample's thickness (25–45 nm) is not known exactly; the C1 values depend on it directly.
- The literature camera length here (~351 mm) is interpolated from the 340 mm calibration point.
- The C1 sign relation between DCORprime and the TEM UI Defocus is not yet verified on this microscope.
- The 300 kV calibration (1.058 mrad/px at 270 mm) disagrees by 1.6× with the 1.689 mrad/px measured from an 80 kV dataset (docs/SITE-FACTS.md §3 in the AutoACEM repository): the nominal camera length does not give the same real camera length at every voltage. Use the calibration for the voltage in use.
- The calibration was measured with a 30 mrad probe; the BF radii at 21.5 mrad are scaled from it.

- Version 2 of this note assumed the literature's disc spanned 2/3 of the detector and recommended 550 mm; the literature's 0.823 mrad/px shows it spans 0.41, so version 3 recommends 430 mm. Version 4 uses the literature's 21 nm thickness: its 0.73 margin sets C1 = +10 nm (version 3 had +15 nm).

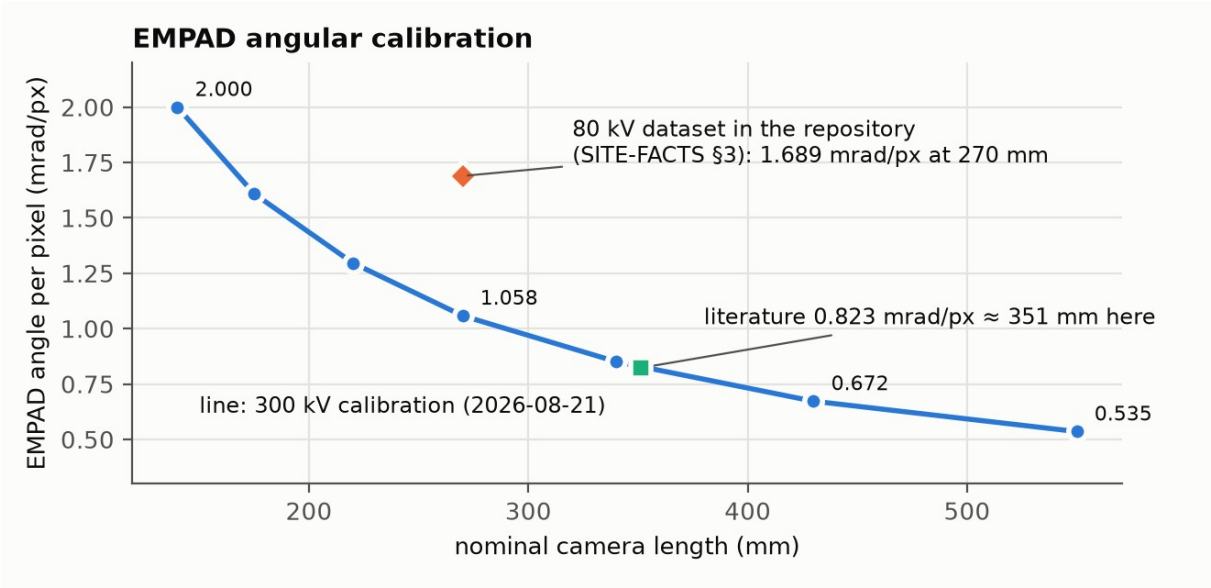


Figure 4. EMPAD angle per pixel vs camera length at 300 kV (calibration in this folder), with the literature's 0.823 mrad/px and the 80 kV value used so far in the AutoACEM repository.

Formulas and inputs

- λ = 1.9687 pm (300 kV); t = 40 nm; α = 21.5 mrad; EMPAD 128 px (64 px from axis to edge).
- Window $W = \lambda / \Delta\theta$; detector edge = $64 \times \Delta\theta$; BF disc radius = $\alpha / \Delta\theta$.
- Probe diameter $D(z) = 2\alpha(C1 + z)$, z measured down from the entrance surface; C1 > 0 = crossover above the sample.
- $C1_{max} = f \cdot W / (2\alpha) - t$, f = ½ (strict) or 1 (loose); overlap = 1 − step / D(0).
- Dose = $I \times \text{dwell} / e / \text{step}^2$ (e/Å², step in Å).