# Powder Measurements on the Neutron Diffractometer E2

# at the Hahn-Meitner-Institute

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## 1. Introduction

With an optimized spectrometer, neutron powder diffraction is a powerful tool to investigate structure and magnetic ordering phenomena in condensed matter. The applications range from solid state physics, chemistry, crystallography, materials science to biology and cover both fundamental and applied research.

An obvious way to use a continuous neutron source such as the 10-MW light-water reactor BER-II efficiently is the application of focusing monochromators and multidetectors. The E2 instrument is based on a linear position-sensitive $BF_3$ counter with 400 wires. It is installed at the radial thermal beam tube R1B of the reactor BER-II.

## 2. Diffractometer description

The monochromator is mounted inside a steel/concrete shielding of 120 cm thickness at a distance of 530 cm from the reactor core. The beam tube can be opened/ closed by a special shutter system .

A saphir-filter is reducing the fast-neutron flux. Three collimations may be used in powder diffraction: The primary collimation $a_1$, between the reactor core and the monochromator, $a_2$ between the monochromator and the sample and $a_3$ in front of the detector. The white beam can be collimated to $a_1$ = 10' , 30' or to the high flux mode 60' (open).

The collimated white beam is monochromatized by changeable monochromators: copper (Cu 220), germanium (Ge 311) and pyrolythic graphite monochromator (PG 002). Normally no secondary collimation $a_2$ is installed, as this results mainly in an intensity loss. However, the best resolution is obtained with $a_1 = 2a_2$ .

The detector system is based on a commercially available $BF_3$ linear position-sensitive counter, covering simultaneously a scattering angle range of 80°, corresponding to 400 detectors with angular separation of 0.2° and a radius of 150 cm. For this detector a special arrangement of anodes/cathodes is used in order to reduce the electronic counting chains (preamplifiers etc.) from 400 down to 40 (20 cathodes and 20 anodes).

The spectra of the different scans are stored on the disk of the instrument workstation CVSE2. Each run may be inspected anytime on a graphic terminal with access to the computer network. The final diffraction pattern is analysed by Rietveld packages on VAX computers. Data are transfered by local area networks. Direct control of running measurements (diffraction pattern, temperature, etc.) or data can be done on all computer in the network.

## 3. Instrumental performance

Two samples (Fe, YAG) are used for the measurement of the instrumental resolution at variable neutron wavelengthes. The experimental results are shown in Fig 1, Fig 2, Fig 3 and Fig 4. In the figures the FWHM corresponds to $\Delta(2\theta)$ and I corresponds to the relative intensity compared with Ge monochromator and 10' collimation.

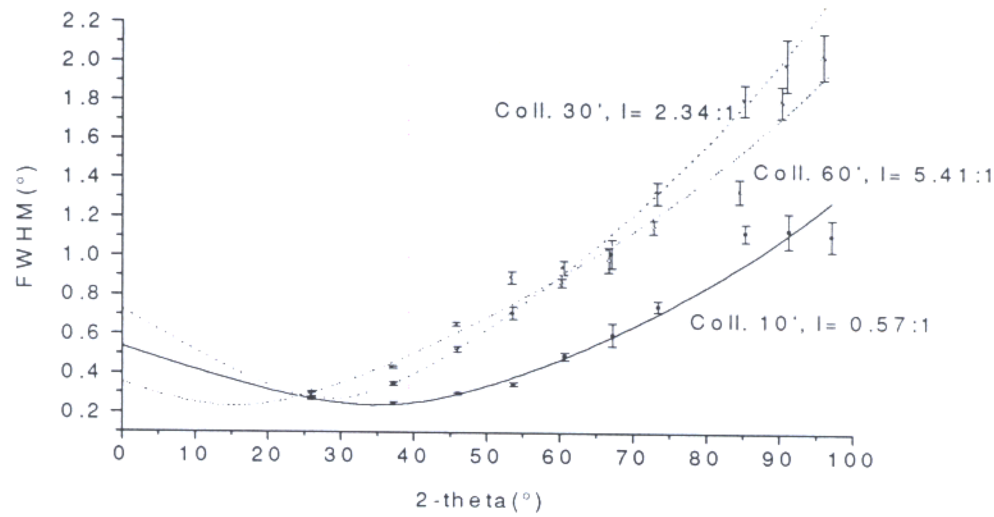



Fig.1: The Resolution of E2 at 0.91A (Mono. Cu. Sam. Fe)

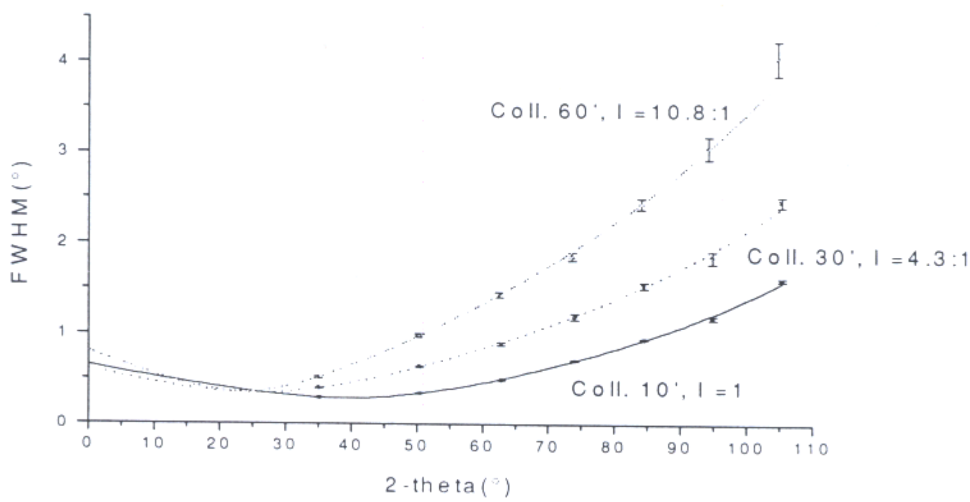



Fig. 2: The Resolution Curve of E2 at 1.216A(Mono.Ge Sam. Fe)

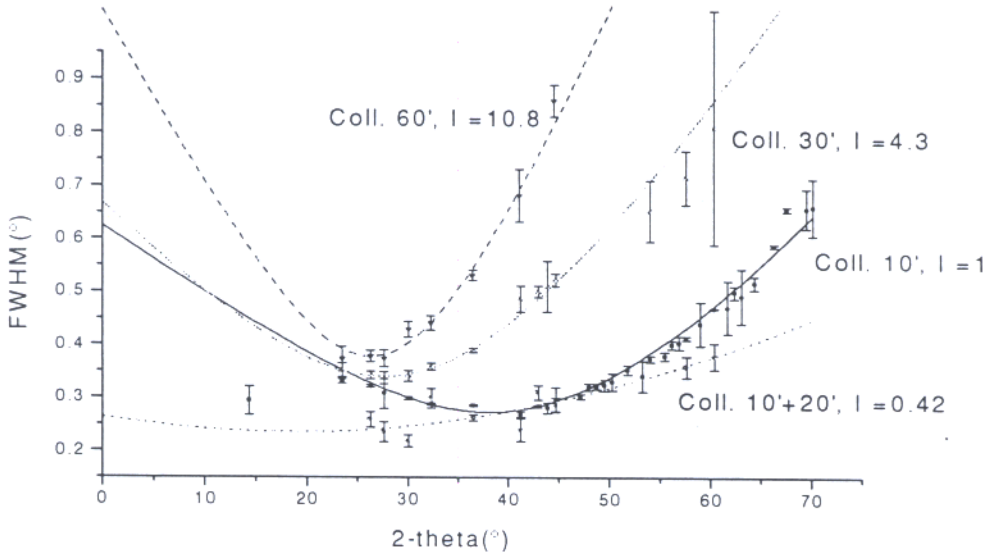



Fig. 3: The FWHM Curve of E2 at 1.2A (Mono.Ge, Sam. YAG)

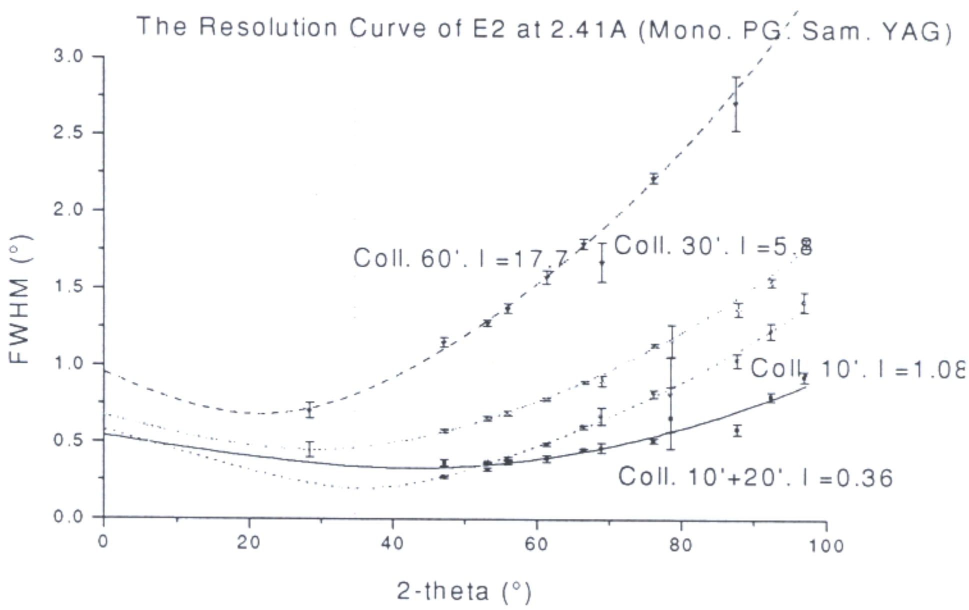



Fig. 4: The FWHM curve of E2 at 2.41A

The fitting function in the figures is of Cagliotti's form:

$$(FWHM)^2 = U tg^2\theta + V tg\theta + W$$

The Δd/d curve of E2 can also be got from experiment. A typical result is shown in Fig. 5.

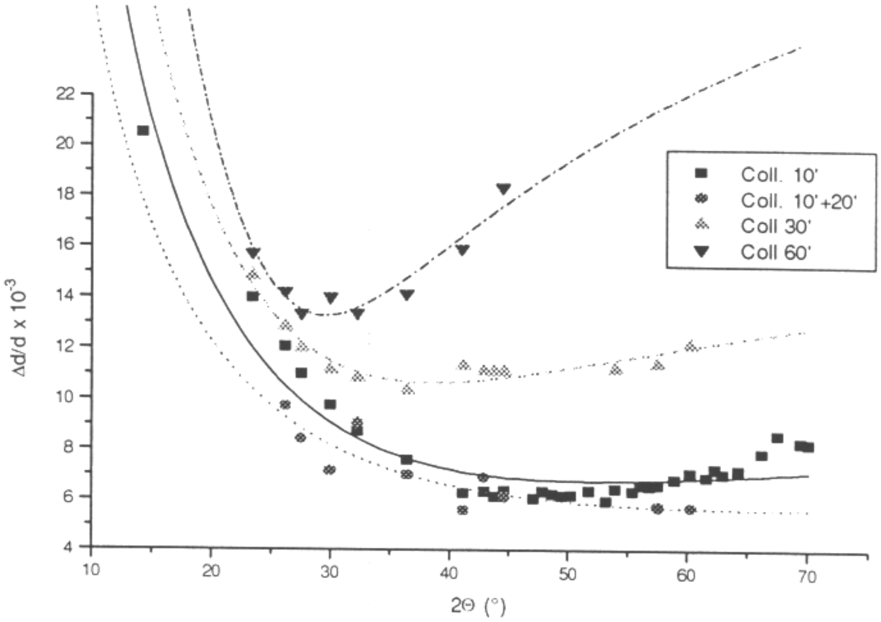



Fig. 5: Δd/d curve at neutron wavelength 1.21A (Ge monochromator)

It is also interesting to compare with the resolutions of different monochromaters. From Fig. 6 we can see that each monochromator has its best resolution region at different d spacings.

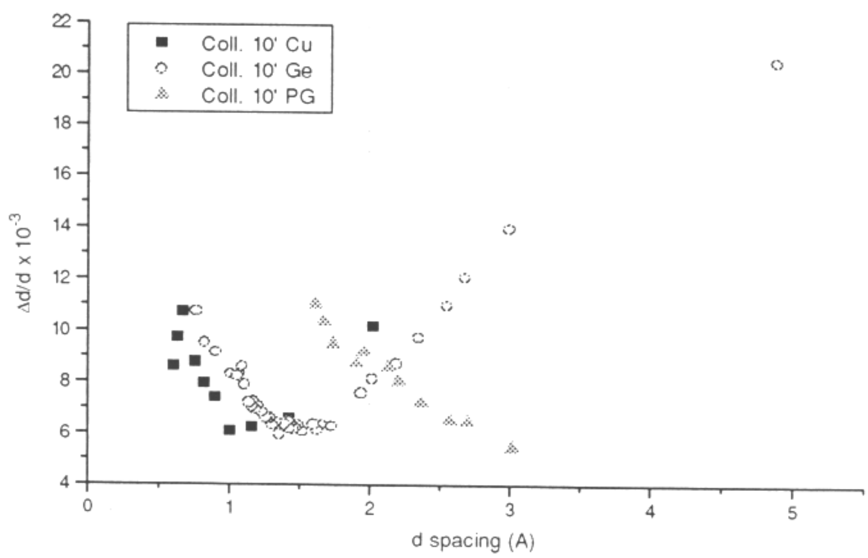



Fig. 6: The resolution of E2 at collimator 10' with different monochromator

3.3. Profile refinement example

Profile refinement has been performed successfully on many samples. As an illustration we show in fig. 7 the result performed recently on YAG.

## 4. Discussion

The instrument E2 installed at the reactor BER-II has good resolution. Without second collimator the minimum value of $\Delta d/d$ is about $6 \times 10^{-3}$. Adding a second collimator (20') can improve the resolution, especially at large diffraction angle, but also reduces the neutrou flux by about a factor 2.

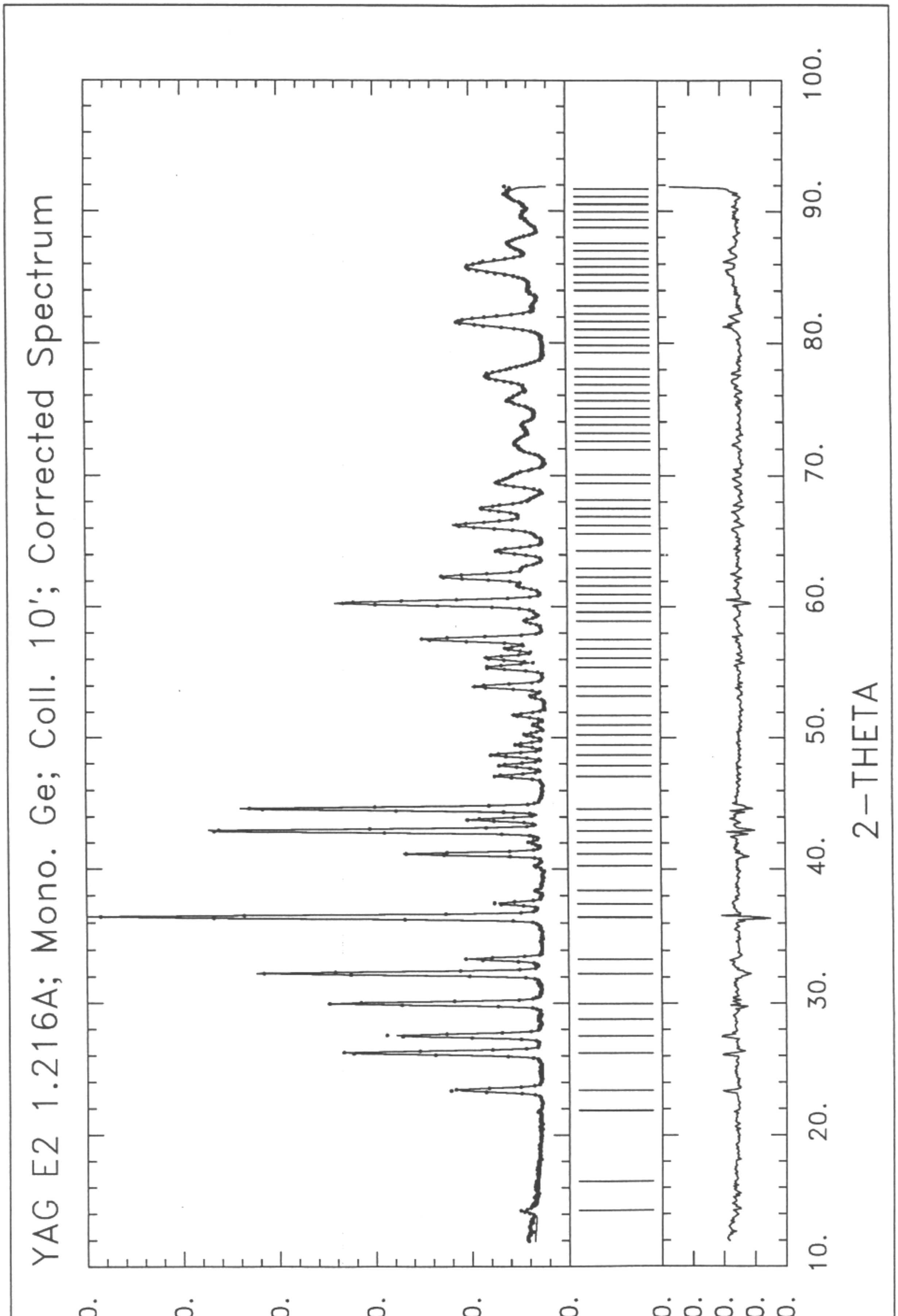
Each monochromato has its best resolution region at different d spacings. The copper monochromator has its best resolution with a first collimator of 10' at a d spacing region below 1Å. Above 2Å of d spacing the pyrolythic graphite monochromator is best. The germanium monochromator is best in the middle range of d spacings.

## Acknowledgements

I appreciate gratefully the Dr. Dietmar Hohlwein, from the group of the Universität Tübingen at the Hahn-Meitner-Institut in Berlin, for great help. I have made this work with his instructions.

I thank also Prof. W. Prandl, Universität Tübingen, and Prof. M. Steiner, BENSC, for providing opportunity to work at the Hahn-Meitner-Institut in Berlin.

Some enlarged figures are shown in the Appendix.



YAG E2 1.216A; Mono. Ge; Coll. 10'; Corrected Spectrum
INTENSITY
25000.
20000.
15000.
10000.
5000.
0.
2500.
1500.
500.
-500.
-1500.
10.
20.
30.
40.
50.
60.
70.
80.
90.
100.
2-THETA


Fig. 7

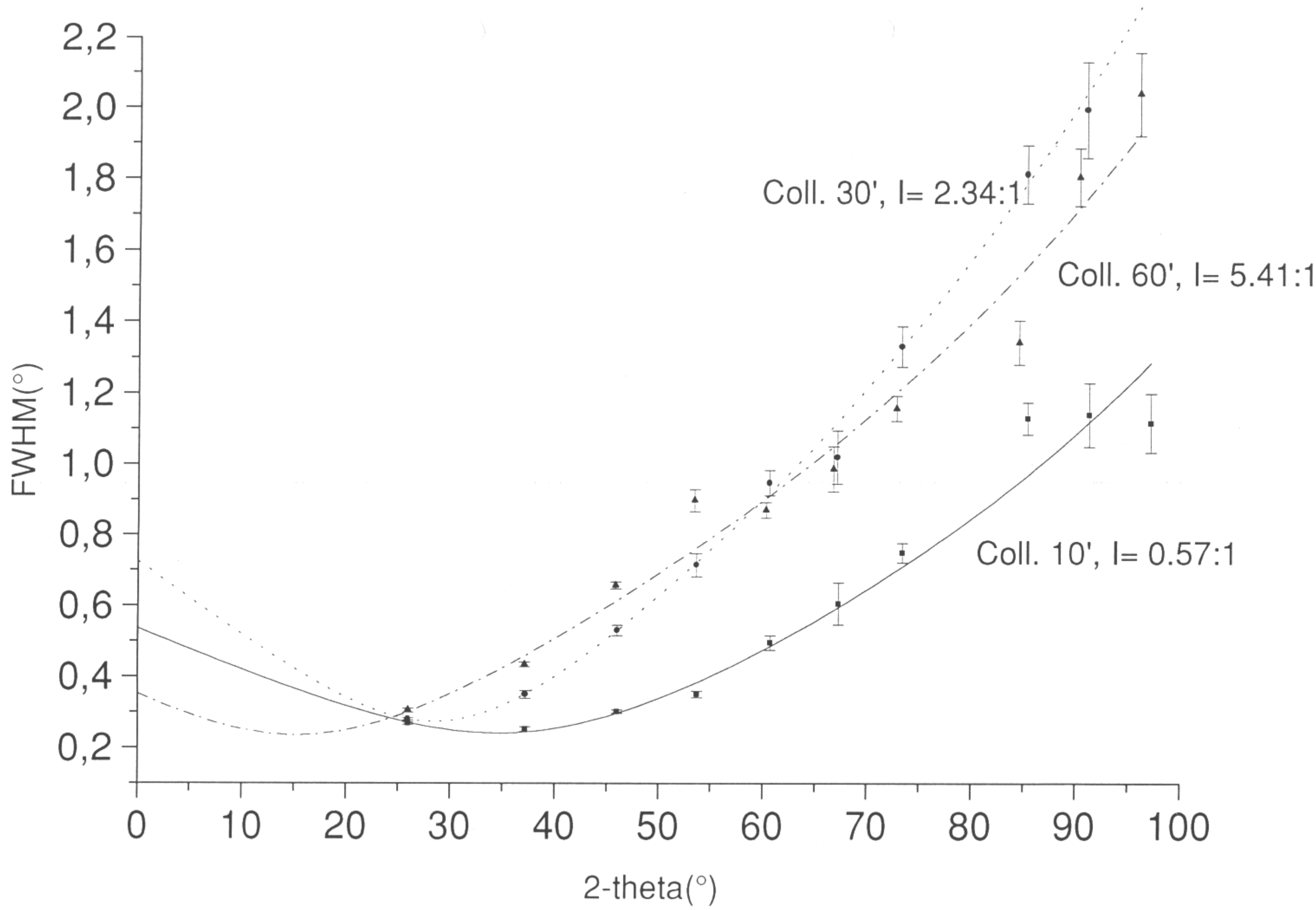



Fig.1: The Resolution of E2 at 0.91A (Mono. Cu. Sam. Fe)

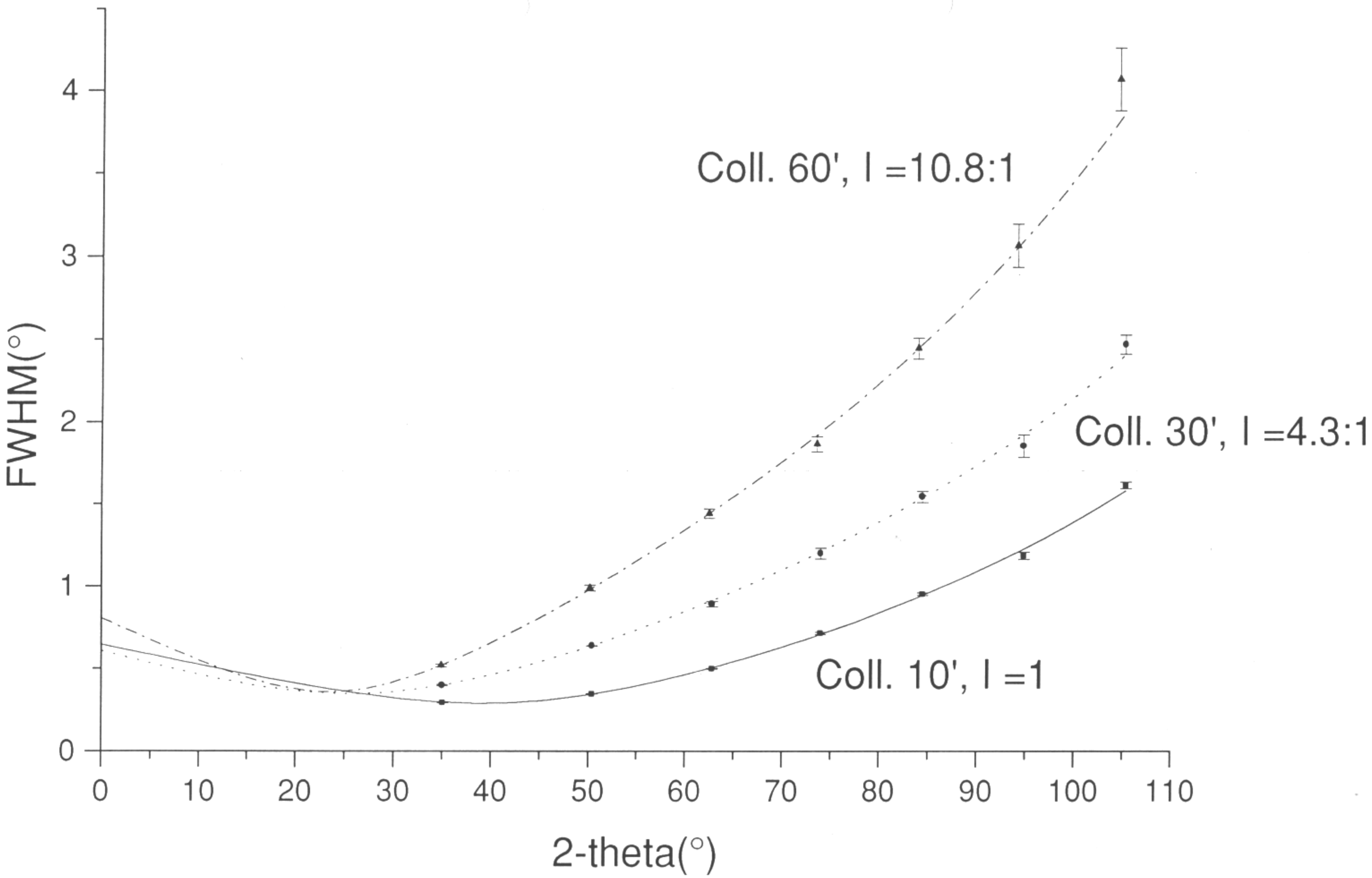



Fig. 2: The Resolution Curve of E2 at 1.216A(Mono.Ge Sam. Fe)

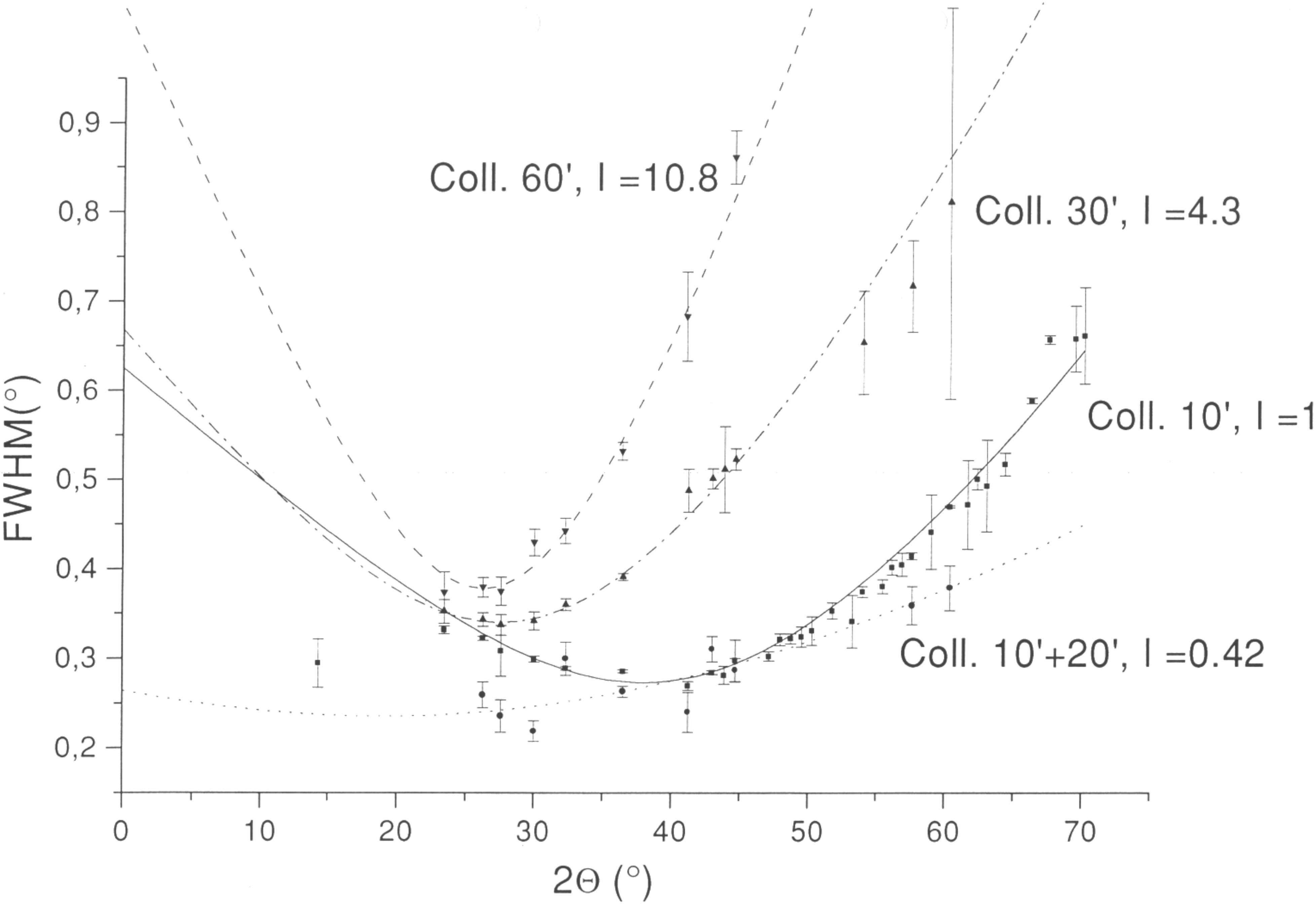



Fig. 3: The FWHM Curve of E2 at 1.2A (Mono.Ge, Sam. YAG)

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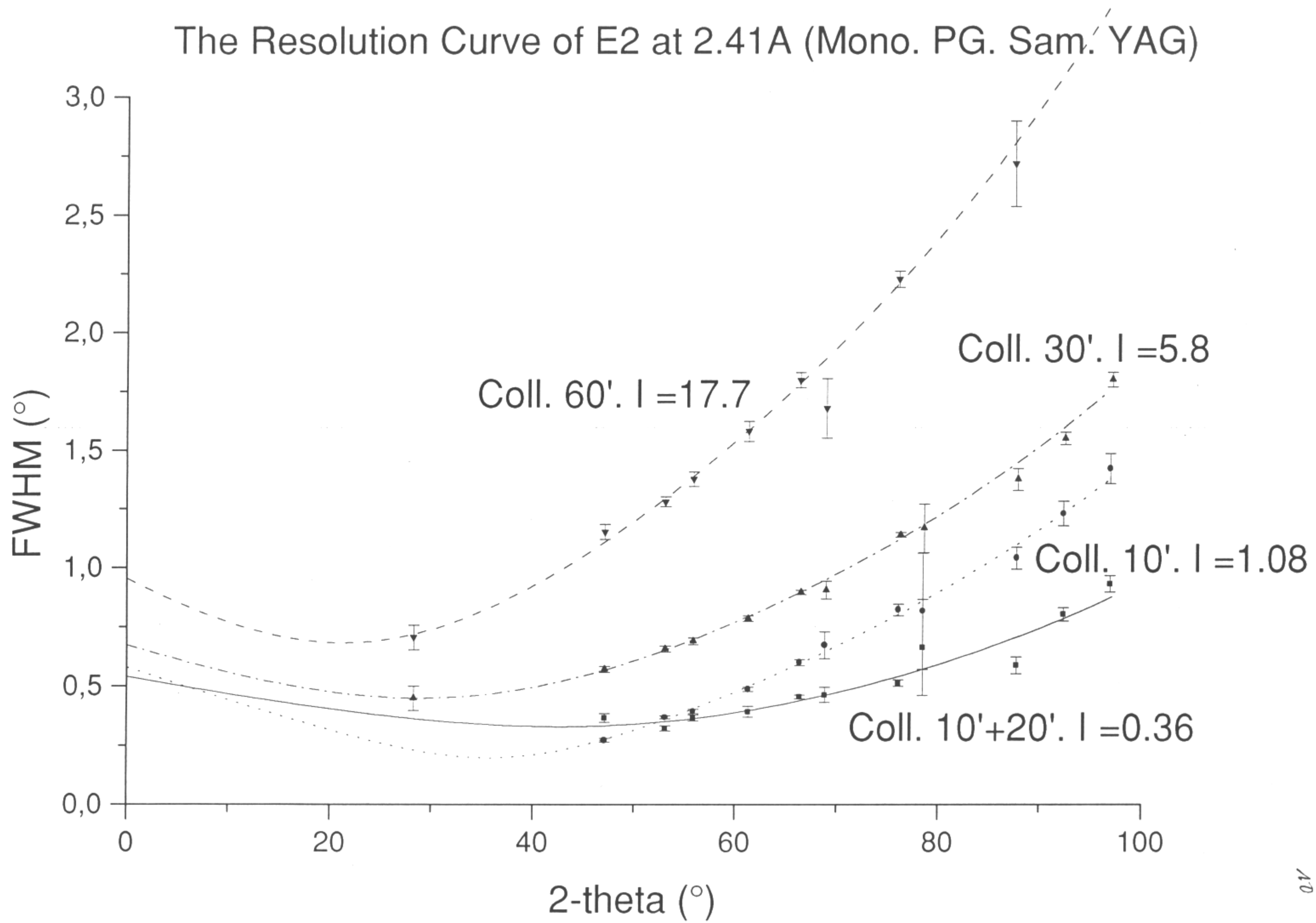
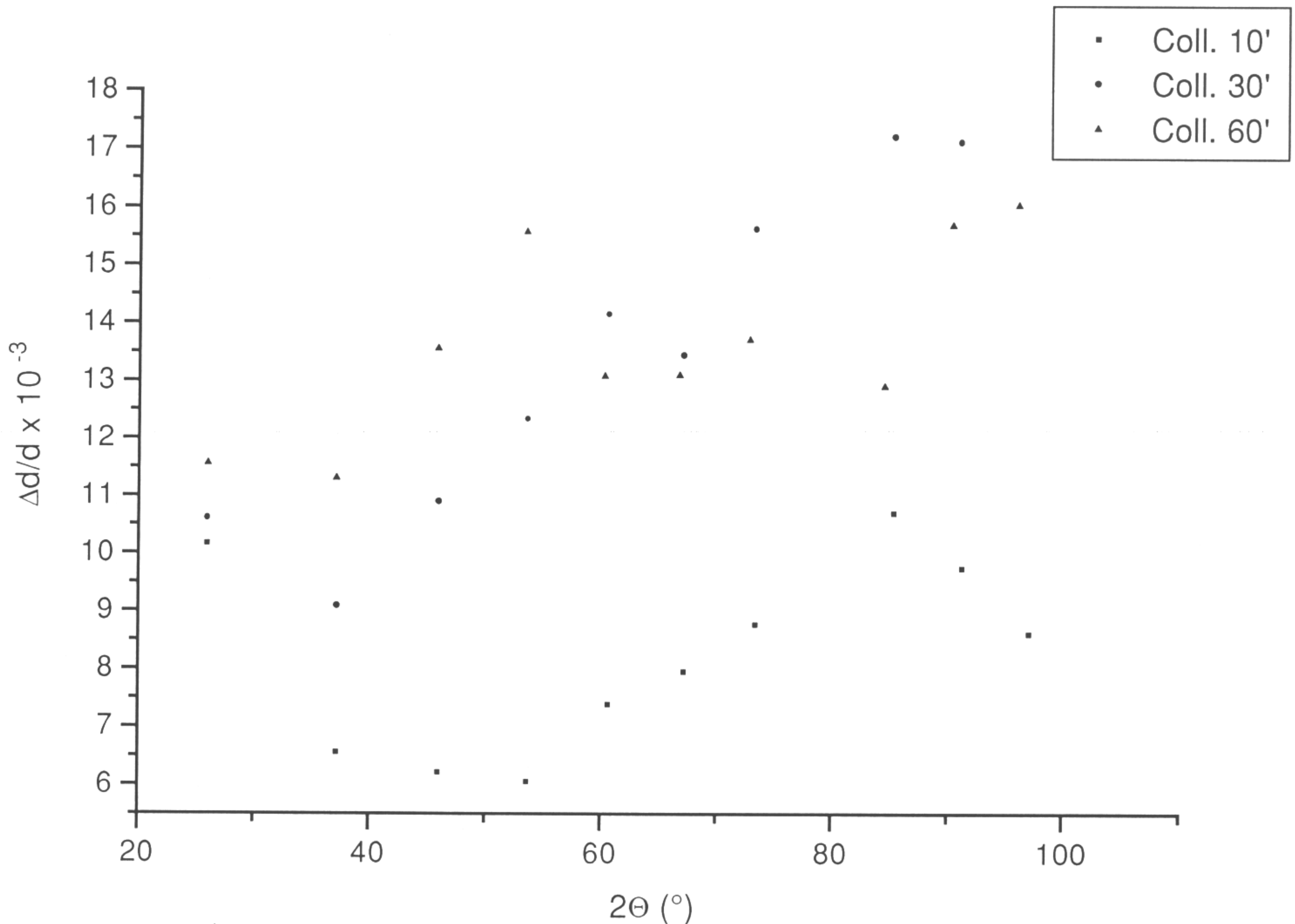
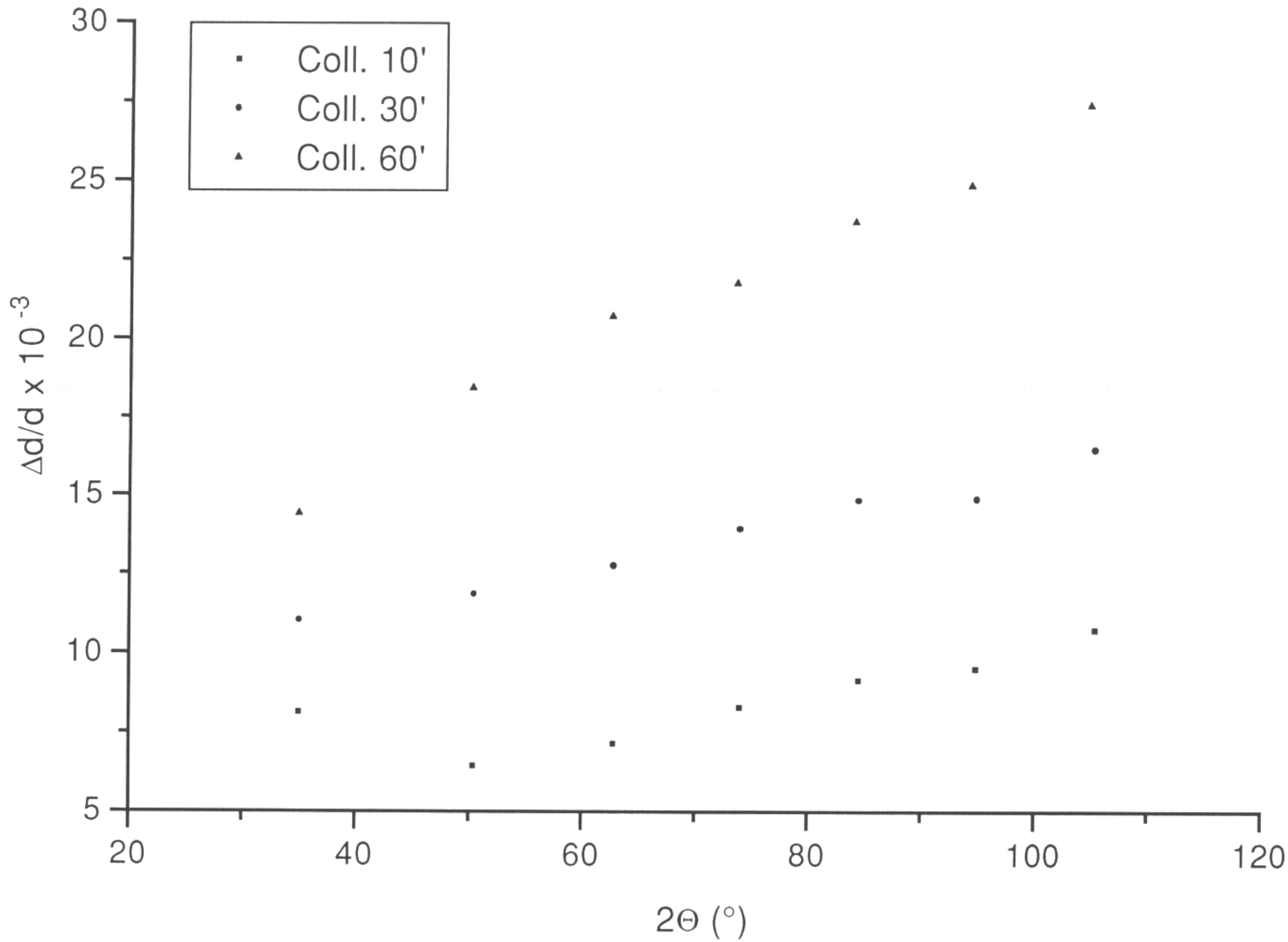
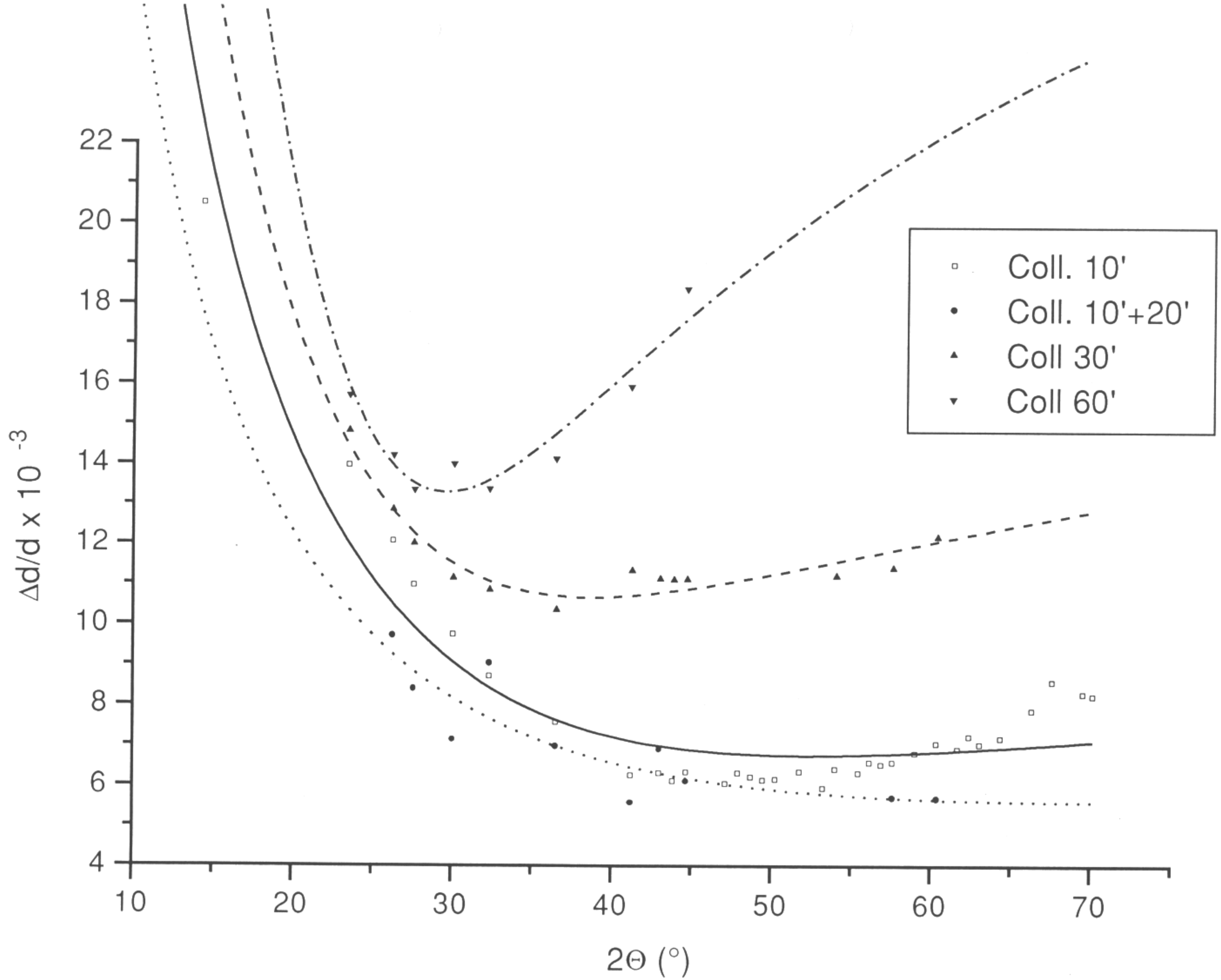

The Resolution Curve of E2 at 2.41A (Mono. PG. Sam. YAG)
FWHM (°)
2-theta (°)
Coll. 60'. I =17.7
Coll. 30'. I =5.8
Coll. 10'. I =1.08
Coll. 10'+20'. I =0.36
3,0
2,5
2,0
1,5
1,0
0,5
0,0
0
20
40
60
80
100



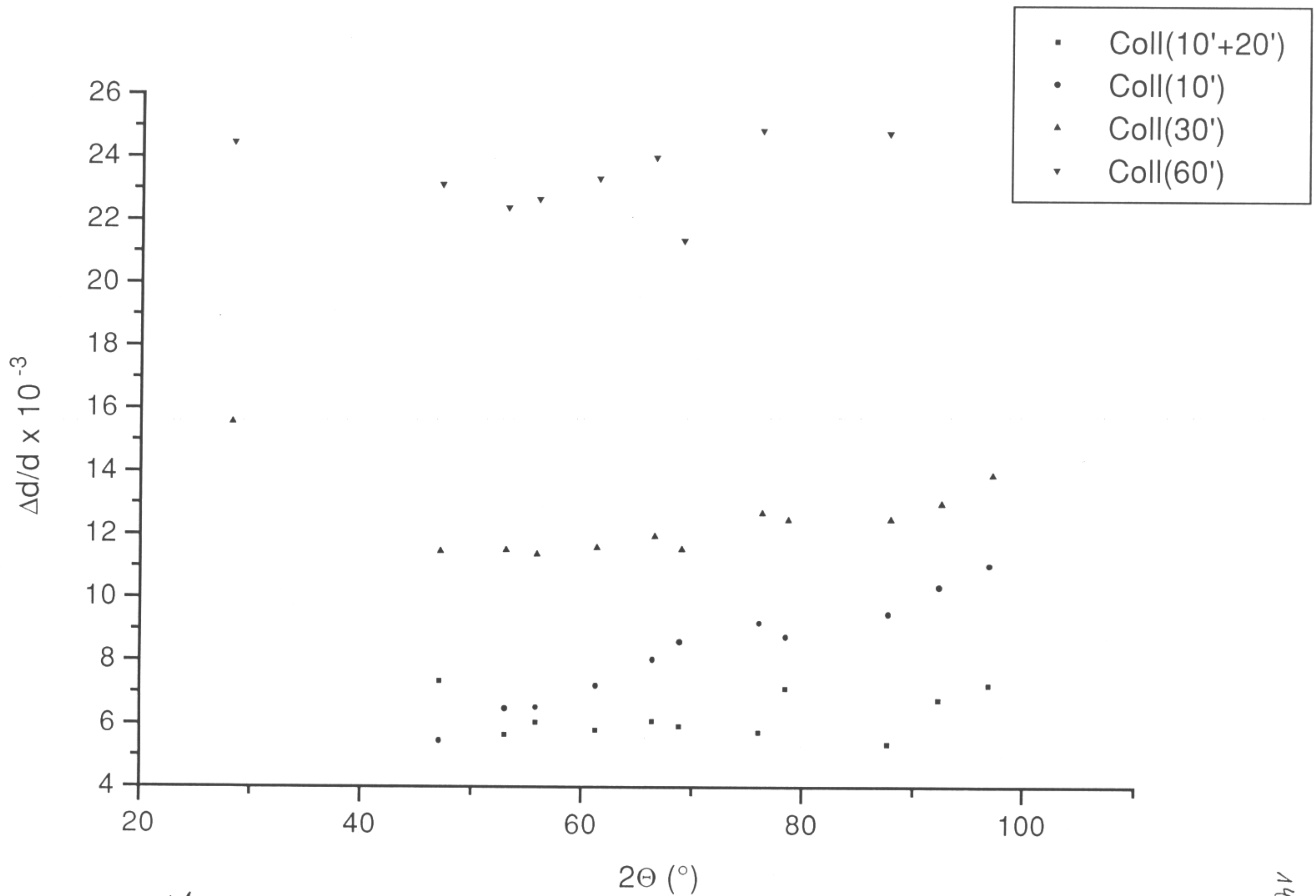

$\frac{\Delta d}{d}$ The Resolution of E2 at 0.91A (Mono. Cu. Sam. Fe)



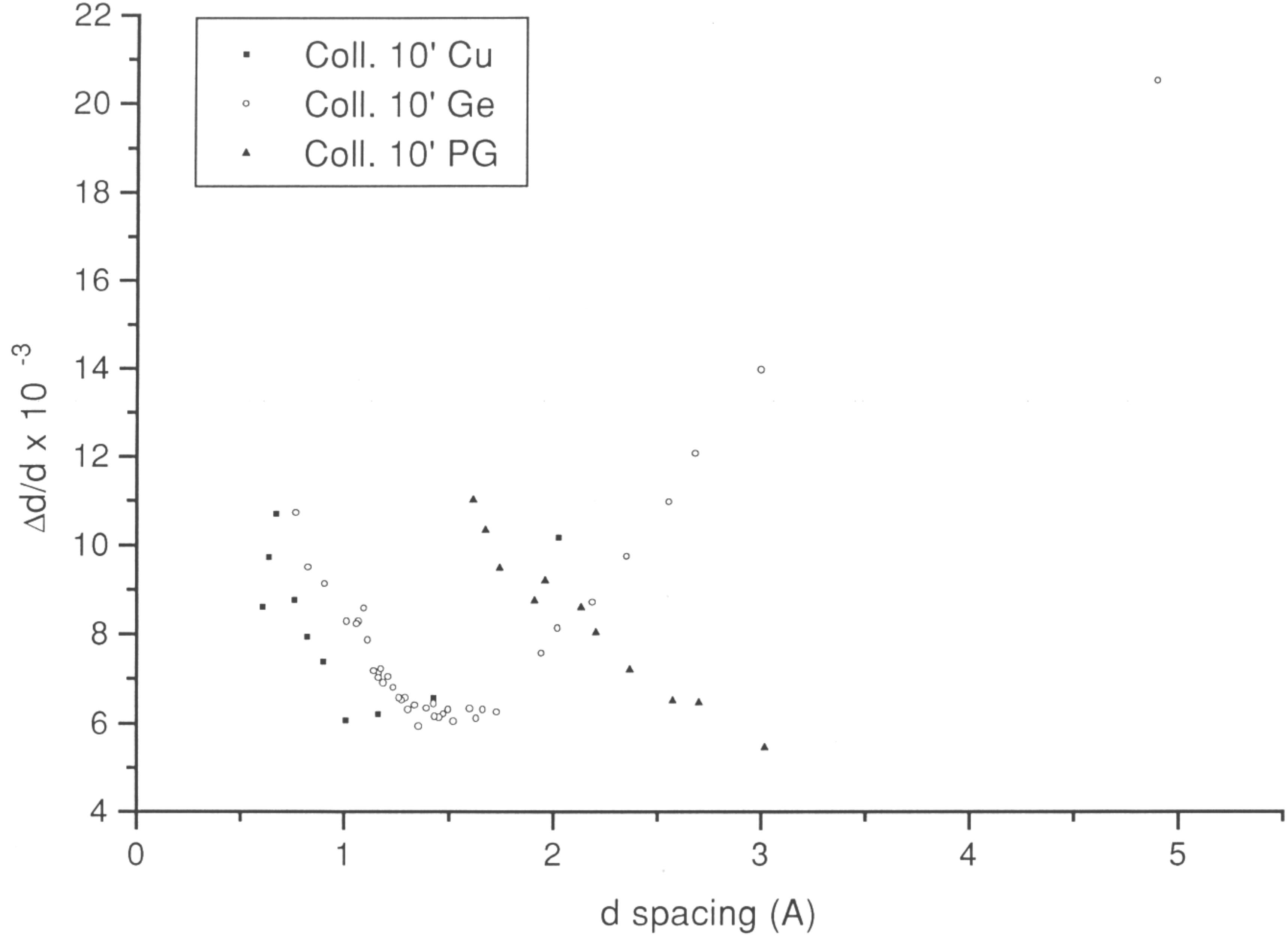

$\frac{\Delta d}{d}$ The Resolution Curve of E2 at 1.216A(Mono.Ge Sam. Fe)

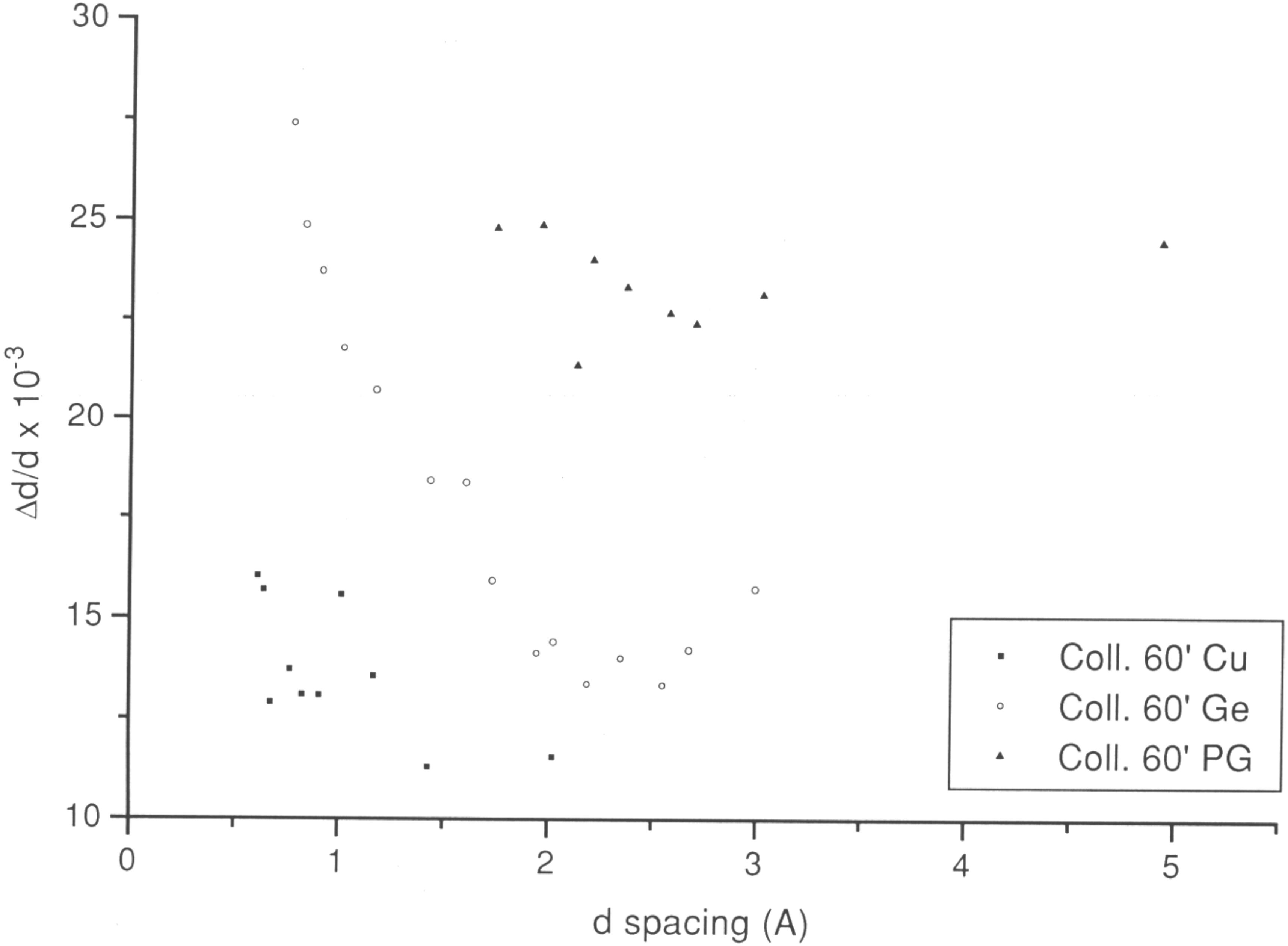



$\frac{\Delta d}{d}$ The FWHM Curve of E2 at 1.2A (Mono.Ge, Sam. YAG)




$\frac{\Delta d}{d}$ The Resolution Curve of E2 at 2.41A (Mono. PG. Sam. YAG)


Coll. 10' Cu
Coll. 10' Ge
Coll. 10' PG
Δd/d x 10 -3
22
20
18
16
14
12
10
8
6
4
0
1
2
3
4
5
d spacing (A)


30
25
20
15
10
Δd/d x 10-3
0
1
2
3
4
5
d spacing (A)
Coll. 60' Cu
Coll. 60' Ge
Coll. 60' PG