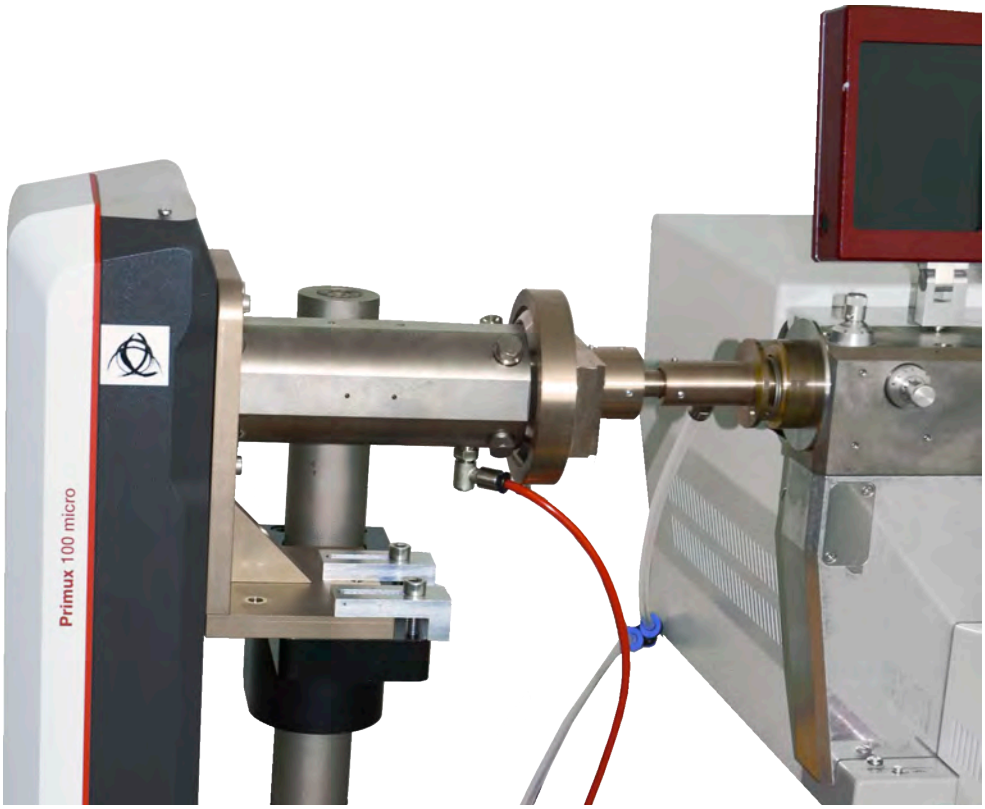


marµX 3G

# Comparing the third generation of micro-beam generators with previous ones

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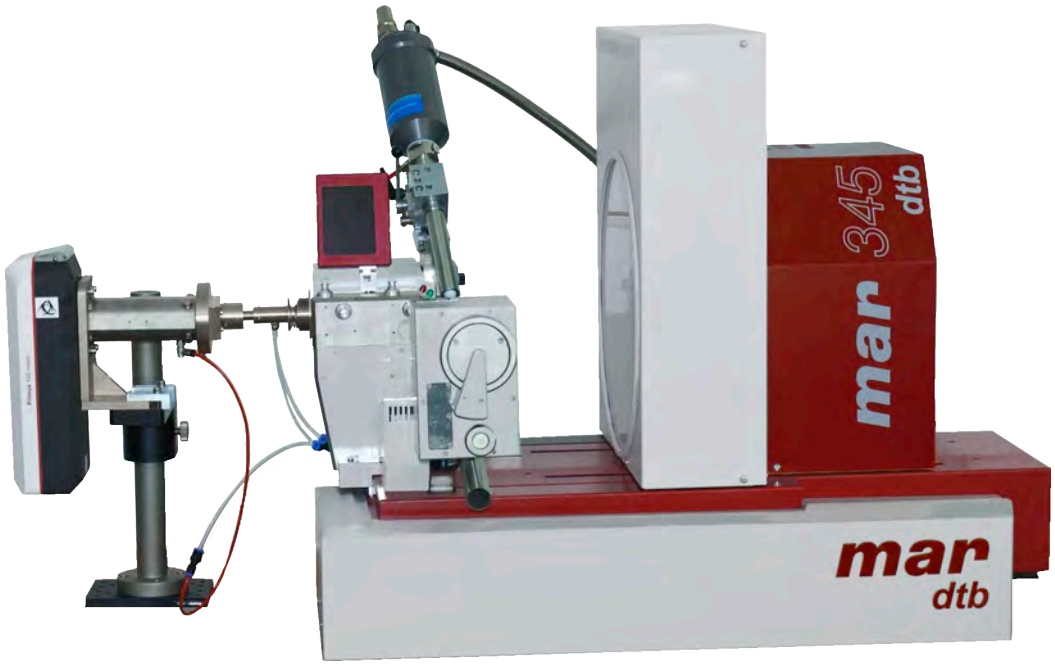
Application Note 15-Aug-18




marμX3G: Primux/ASTIX++ with mar345 detector and mardtb goniostat

# 1. Introduction

The marμX3G system is a very compact yet powerful complete X-ray setup suitable for both protein and small molecule data collection as well as other types of experiments. The marμX3G system consists of:

- the new 2018 model of the Primux 100 Cu X-ray source manufactured by Anton Paar, Austria
- the ASTIX++-f-105/395-L150 multi-layer optics manufactured by AXO, Germany
- a mar345 image plate detector or a DECTRIS PILATUS3 R 1M/300K/200K detector or a DECTRIS EIGER R 4M/1M detector
- a mardtb goniometer system
- an experimental table with an (optional) radiation enclosure
- an (optional) Oxford Cryosystems 800 cooler
- an (optional) marLiN$_2$ automatic LN2 refill system

The Primux 100 source is a new generation of micro-beam X-ray generators that is operated at 50 Watt (50 kV, 1 mA) and produces a spot of 50 µm size on the anode. It is coupled to the latest generation of ASTIX++ high brilliance & high flux multi-layer optics by AXO which comes with a 15 cm confocal mirror which yields a spot of approx 160 µm size at the focal distance of 395 mm with a divergence of 7.5 mrad and a highly symmetric beam profile. The combination gives an X-ray data collection with unprecedented brilliance and flux that matches the one from rotating anode micro-focus generators like the Rigaku 007 - but without the hassles and costs that are typical for maintaining rotating anodes. In fact, the Primux 100 system is virtually maintenance free and you can expect it to run for a couple of years without ever creating maintenance issues besides occasionally checking the cooling water of the closed-circle air/water-cooler.

In this study we compare the new marμX3G system based on the Primux/ASTIX++-source with the previous generations in particular:

- the marμX2G system with an Incoatec IμS generator and optics operated at 30 W (50 kV / 0.6 mA).
- the marμX1G system with a GeniX 3D generator and optics operated at 30 W (50 kV / 0.6 mA).

In order to asses the quality of the new system, several small molecules of different types and sizes have been collected. Here we present a selection of typical results.

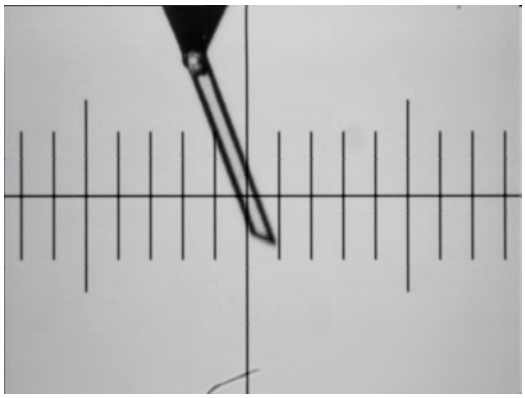
# 2. Data collection

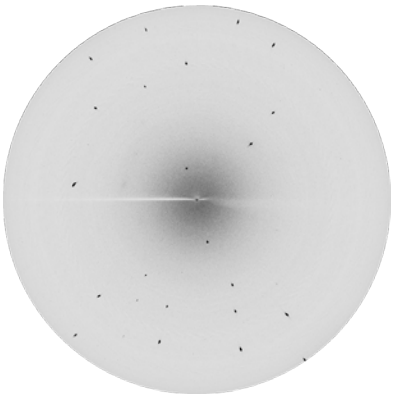
Data for all crystals were collected on several mar345dtb detector systems. However, calibration procedures yield the same response for the detectors within 2% accuracy. On all mardtb goniostats, the beam was carefully aligned. For data collection, the slits of the collimator were left wide open to prevent any cutting down of the size of the native primary beam. The crystals used for data collection were exactly the same. They do not suffer from degradation and alteration over time, so even if the measurements are effectively years apart (Genix in 2010, IμS in 2015, Primux in 2018), the diffraction results rather reflect the quality of the beam and crystal effects should be negligible.
Data were collected at room temperature and processed using the automar program suite.

## 2.1 Ammonium bitartrate 1

Data collection:

- Crystal size: 30 x 40 x 700 μm
- Space group: $P2_12_12_1$
- Cell axes: 7.6 x 7.8 x 11.6 Å
- Oscillation range: $360^0$
- $5^0$/image
- 1.51 Å resolution
- Multiplicity: 11.5
- Completeness: 97.8%





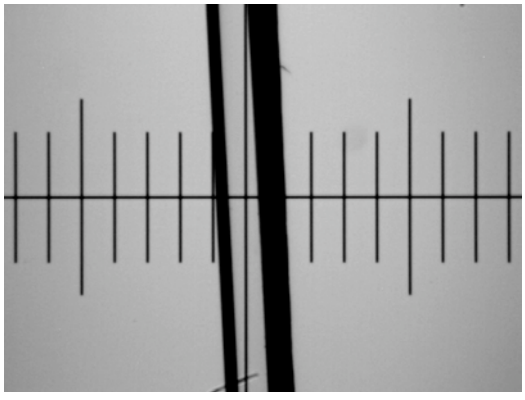
| | | Primux | IμS | GeniX |
|---|---|---|---|---|
| **Exposure time/image** | **[sec]** | 16 | 16 | 16 |
| **# unique reflections**[1] | | 135 (18) | 135 (18) | 135 (18) |
| **Rmeas**[1] | **[%]** | 2.6 (3.5) | 3.6 (3.0) | 3.03 (5.66) |
| **<Intensity/σ>**[1] | | 63.1 (66.1) | 55.1(55.7) | 18.5 (10.5) |
| **<Intensity>** [1] | | 76727 (18126) | 47448 (12105) | 29028 (6723) |
| **Relative <Intensity>** | | **2.64<br>1.61** | **1.63<br>1.0** | **1.0<br>0.61** |

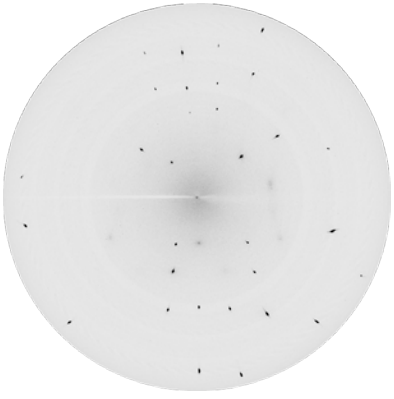
[1] Last shell in brackets: 1.51-1.56 Å

## 2.2 Ammonium bitartrate 2

Data collection:

- Crystal size: 220 x 150 x 1200 μm
- Space group: $P2_12_12_1$
- Cell axes: 7.6 x 7.8 x 11.6 Å
- Oscillation range: $360^0$
- $5^0$/image
- 1.51 Å resolution
- Multiplicity: 11.5
- Completeness: 97.8%





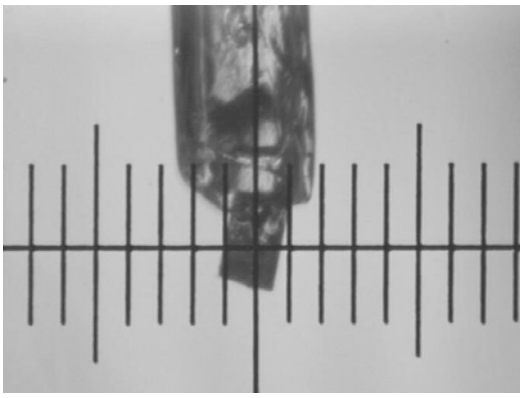
| | | Primux | IµS | GeniX |
|---|---|---|---|---|
| **Exposure time/image** | [sec] | 8 | 8 | 8 |
| **# unique reflections**[1] | | 126 (18) | 127 (18) | 127 (17) |
| **Rmeas**[1] | [%] | 3.74 (4.96) | 4.51 (2.22) | 3.03 (2.77) |
| **<Intensity/σ>**[1] | | 47.7 (38.3) | 49.5 (58.0) | 58.6 (56.8) |
| **<Intensity>** [1] | | 246582 (91976) | 200254 (79889) | 94725 (28508) |
| **Relative <Intensity>** | | **2.60<br>1.23** | **2.11<br>1.0** | **1.0<br>0.47** |

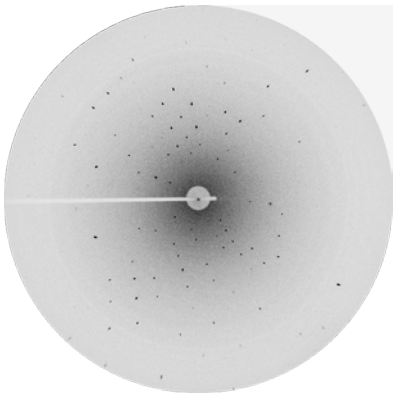
[1] Last shell in brackets: 1.51-1.56 Ang.

### 2.3 Small molecule $C_{18}H_{46}Cl_3CoN_6O_2$

Data collection:

- Crystal size: 120 x 30 x 150 µm
- Space group: I-42d
- Cell axes: 19.0 x 19.0 x 14.0 Å
- Oscillation range: $150^0$
- $5^0$/image
- 1.51Å resolution
- Multiplicity: 4.90
- Completeness: 99.8%





| | | Primux | IµS |
|---|---|---|---|
| **Exposure time/image** | [sec] | 4 | 4 |
| **# unique reflections**[1] | | 419 (29) | 417 (31) |
| **Rmeas**[1] | [%] | 6.8 (13.2) | 7.0 (13.8) |
| **<Intensity/σ>**[1] | | 6.8 (4.1) | 6.2 (4.1) |
| **<Intensity>** [1] | | 18207 (7805) | 8329 (3759) |
| **Relative <Intensity>** | | **2.18** | **1.0** |

[1] Last shell in brackets: 1.50-1.55 Ang.

## 3. Conclusion

The average intensities from data processing of the collected data sets are a meaningful comparator provided that the diffraction power of the samples stays constant over time and that the gain of the detectors used for the experiments is constant. The average intensities then show how many X-rays are effectively seen by the sample and produce diffraction. From the data here we deduce, that the new Primux/ASTIX-source is a significant improvement over previous generations of sealed-tube micro-focus generators and can yield 2 times more X-rays as compared to the IuS-source. The ratio seems to become more favourable for the Primux source for very crystals, indicating a much higher brilliance, i.e. a concentration of X-ray intensity in a small spot.