Application Note SC-XRD 515

What's in those sweetener packages anyways?

- Fast screening of organic compounds with the large PHOTON II detector, the bright IμS 3.0 microfocus source and APEX3's powerful XPRESSO routine

Introduction

This application note demonstrates fast screening of several crystals from a natural sweetener packet. The very intense IμS 3.0 microfocus source and the large PHOTON II allow collecting complete data to about 1 Å in 3 min. These fast data sets all processed using the APEX3 software suite allow comprehensive quality assessments as well as preliminary structure determinations.

How Sweet is Sweet?

Times have certainly changed. Years ago, there were only three choices for sweetening your tea or coffee: white sugar, brown sugar, or honey. Today, with all the attention on calorie, sugar, and carbohydrate intake, many people do not even consider those traditional sweeteners anymore. Instead, numerous sugar substitutes are available, giving consumers the choice between the blue packet

"Equal" (aspartame), yellow packet "Splenda" (sucralose), or pink packet "Sweet'n low" (saccharin). And now there's a relatively new calorie-free sugar substitute available served in a green packet "Truvia" (stevia) (Figure 1).

Figure 1: Colorful collection of sweetener packets

Crystals from the Green Packet

Opening a green package of Truvia revealed nicely formed colorless crystals, but a glance at Truvia's list of ingredients shows Erythritol ($C_4H_{10}O_4$) as the main ingredient (Figure 2) and second on the list is Rebiana, high-purity rebaudioside A ($C_{44}H_{70}O_{23}$), a steviol glycoside (Figure 3). A quality check on the crystals was used to quickly reveal the structure of the molecules forming the colorless crystals.

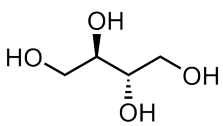

Figure 2: Erythritol, a sugar alcohol.

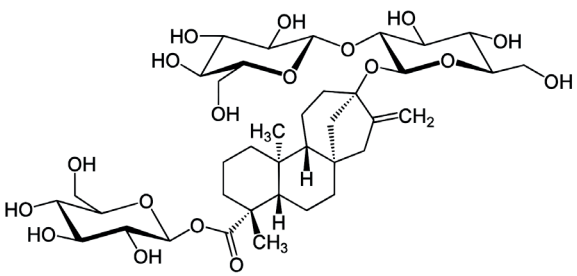

Figure 3: Rebaudioside A, a stevioside with steviol as the central building block.

Experimental

Preparation

Four approximately isometric crystals were pre-selected from the bulk material, all with dimensions of around 200 micrometers. Clear crystals were chosen that did not appear to be split or twinned when viewed at 40-fold magnification. Crystals were mounted on a Mitegen mount covered in a thin film of mineral oil. After the initial quality check crystals were removed from the goniometer and stored in a numbered position on a microscope slide for further experiments if needed.

Instrumentation

All crystals were investigated consecutively with a D8 VENTURE with KAPPA goniometer, IμS X-ray source equipped with a Cu-tube, and PHOTON II detector. Even with little experience, crystals can be mounted and centered in less than one minute. Crystals were screened at 100K using a Fast Scan, collecting 180˚ of data in 3 min (1˚/s).

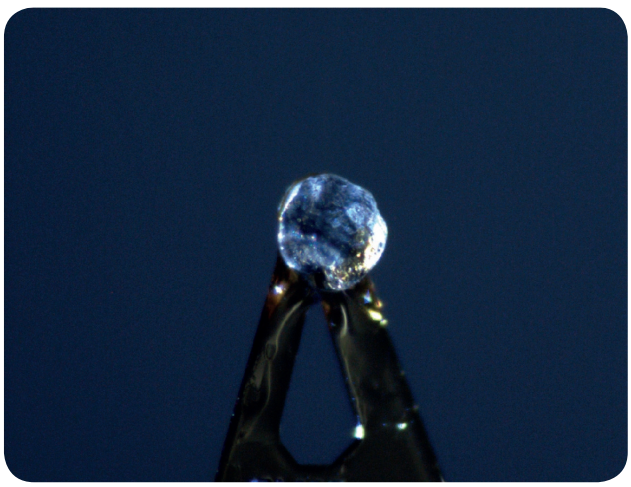

Figure 4: Video image of loop-mounted Crystal 3.

At a short sample-detector distance of 40 mm the large PHOTON II covers a range of >105˚ data in 2θ, which corresponds to a resolution of about 1 Å. A single scan of 180˚ of data to 1 Å resolution typically provides sufficient data not only for quality assessment but also for initial structure determination.

Quality Check

In APEX3 the quality check is automated and includes harvesting reflections from the data scan, indexing, Bravais check, cell refinement as well as a search in the optional Cambridge Structure Data base (CSD).

Although a unit cell was successfully determined for crystal 1 only about half of the reflections could be indexed (Figure 5) suggesting that the crystal has quality problems. The APEX3 suite's advanced software tools allowed the detailed investigation of the problems, which, however, is beyond the scope of this application note.

For crystal 1 the Bravais lattice type was determined as Tetragonal I and a CSD cell search found 14 matching unit cells. Four entries with the code MERYOL are good matches with a sum formula of $C_4H_{10}O_4$ strongly suggesting that the crystal is in fact Erythritol (Figure 6) and not Rebiana.

☑	Index	Selected 1181 reflections for indexing.
☑	Choose Solution	Indexed 681 out of 1181 reflections.
☑	Refine	6.78 9.60 9.61 82.75 69.50 68.93
☑	Bravais	Found Bravais lattice type Tetragonal I.
☑	Refine	12.71 12.71 6.77 90.00 90.00 90.00
☑	Search	Found 14 matching unit cells.

Figure 5: Results of quality check for Crystal 1

Identical to crystal 1, data of crystals 2 to 4 were also processed using XPRESSO and for all remaining samples the data quality was sufficient to obtain an initial structure. Figure 8 provides an approximate timeline for the screening experiment and illustrates that APEX3 enables to conveniently screen four crystals including initial structure determination in less than 30 minutes. Based on the lowest R1 reliability criterion crystal 3 was chosen as the best crystal and re-mounted for a full data collection to 0.8 Å.

Unit Cell: a=12.65Å, α=90.00°, V=1077Å³
b=12.65Å, β=90.00°, Tetragonal I
c= 6.73Å, γ=90.00°

a [Å]	b [Å]	c [Å]	α [°]	β [°]	γ [°]	Space Group	Formula	Identifier
12.83	12.83	6.59	90.00	90.00	90.00	I -4	C24 H20 Sb1 1+,O4 Re1 1-	FUSGUW
12.57	12.57	6.80	90.00	90.00	90.00	I -4	C24 H20 Sb1 1+,Cl1 O3 1-	FUSHAD
12.67	12.67	6.71	90.00	90.00	90.00	I -4	C24 H20 Sb1 1+,Cl1 O4 1-	GAVLIY
12.81	12.81	6.81	90.00	90.00	90.00	I 41/a :2	C4 H10 O4	MERYOL
12.80	12.80	6.84	90.00	90.00	90.00	I 41/a :2	C4 H10 O4	MERYOL01
12.81	12.81	6.81	90.00	90.00	90.00	I 41/a :2	C4 H10 O4	MERYOL02
12.71	12.71	6.75	90.00	90.00	90.00	I 41/a :2	C4 H10 O4	MERYOL03
12.55	12.55	6.85	90.00	90.00	90.00	I -4	C24 H20 As1 1+,I1 O4 1-	SOVYIL
12.80	12.80	6.85	90.00	90.00	90.00	I -4	C4 H6 F2 O2	ZZZUNI

Figure 6: Cell search results for crystal 4

Full structure determination

For the full structure determination, crystal 3 was remounted. A complete data set with an average redundancy of about 5, to a resolution of 0.8 Å was acquired in 80 min. Given the industrial crystallization process, data are of excellent quality with an Rsym of 1.97% and an Rsigma of 1.50%. The reliability criterion R1 for the structure is 2.50%. The crystal structure is a 3-D network of hydrogen-bonded Erythritol molecules. Electron density supports disordered hydrogen positions of the hydroxyl groups. (Figure 7).

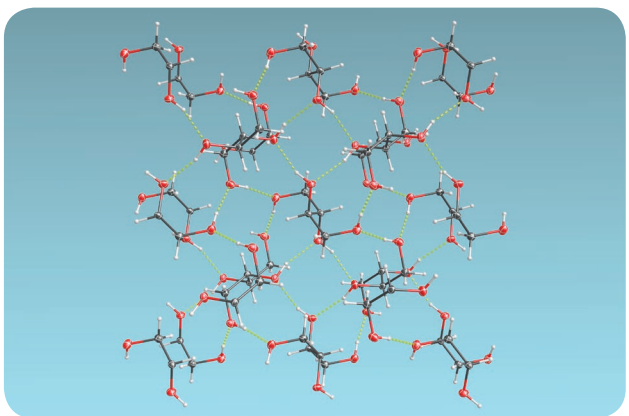

Figure 7: Hydrogen-bonded network of Erythritol molecules.

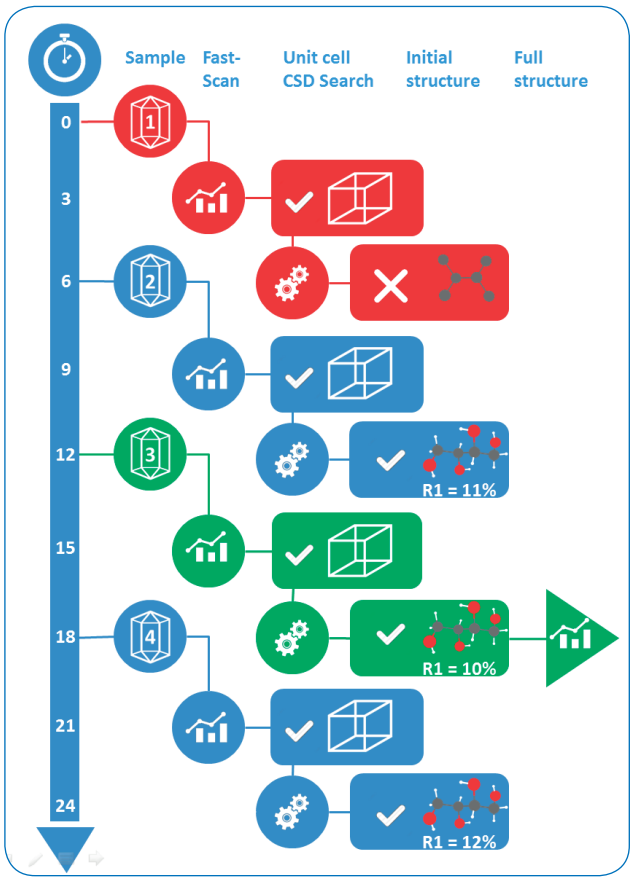

Figure 8: Timeline for screening four crystals in less than thirty minutes

Conclusion

- D8 VENTURE equipped with the PHOTON II detector and Cu IμS 3.0 X-ray source provides a powerful system that allows fast screening of samples in minutes even from the longer wavelength Cu-radiation.
- The detector's solid angle is large enough to allow data acquisition to 1 Å in one detector setting. The powerful Cu IμS 3.0 X-ray source provides diffraction data from smaller samples. The sensitive and shutterless PHOTON II detector delivers dead-time-free screening and full data collection.
- The bittersweet result of this application note is that all crystals investigated appear to be nothing more than sugar-alcohol Erythritol crystals.

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