ESRF	**Experiment title:** Dynamic and thermodynamic properties of glycine polymorphs from simultaneous analysis of multi-temperature diffraction data	**Experiment number**: CH-3070
Beamline: ID11	**Date of experiment**: from: 01st July 2010 at 08h00 to: 04th July 2010 at 08h00	**Date of report**: 28/02/2011
Shifts: 9	**Local contact(s)**: Loredana Erra	*Received at ESRF:*

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Report:

The research project has a three-fold aim: i) measuring accurate atomic displacement parameters (ADPs) of glycine polymorphs (α, β and γ) from 10 to 298 K, ii) investigating the dynamical properties of glycine molecules in the crystals, and iii) determining the thermodynamic parameters (specific heat and entropy).

Synchrotron data to 0.5 Å resolution at 10, 108, 130, 190, 250 and 300 K for the α-glycine polymorph and at 10, 108, 200 and 300 K for the γ-glycine polymorph were collected at beamline ID11 with a wavelength of 0.15927 Å, using a homemade CCD, 3-circle diffractometer. Data were processed with SAINT+, giving completeness = 98%, redundancy = 9.8–25.6, R_{int} = 0.034–0.043, and the structures were refined with SHELXL-97, yielding R_1 = 0.025–0.050, $|\Delta\rho|$ = 0.22–0.50 e Å^{-3}, EXTI = 0.000 (Table 1). In the overall statistics, the data are of good quality. Residual electron density peaks are observed on the bonds as expected and in addition at the atoms of γ-glycine at 300 K (Fig. 1b). The ADPs of α- and γ-glycine behave normally and agree with those from other sources (Fig. 2).

The normal mode analysis [1] of all the data available is on going. The preliminary analysis using a rigid body model of motion gives external vibration frequencies in fair agreement with the spectroscopic data: 70.3, 78.0, 86.5, 125, 136 cm^{-1} vs. 52, 73, 109, 162, 190 cm^{-1} [2] for the α-form and 56.5, 70.1, 73.3, 130 cm^{-1} vs. 89, 104, 138, 152 cm^{-1} [3] for the γ-form. The external frequencies from the ADP analysis together with the internal frequencies from high-level ab initio calculations will be used to estimate the thermodynamic parameters.

Table 1. Data statistics of α- and γ-glycine from beamline ID11

T (K)	Compl	Redund	R_{int}	Reflns All/Obs	R_1	Δρ (e Å^{-3})	Δρ positions	EXTI
10	98.1	12.64	0.043	2290/2289	0.025	0.47/–0.31	@ bonds only	0.00000
108	97.8	14.09	0.040	2290/2281	0.029	0.50/–0.33	@ bonds only	0.00000
130	97.6	12.59	0.037	2289/2286	0.029	0.49/–0.30	@ bonds only	0.00000
190	97.7	12.45	0.035	2315/2302	0.032	0.43/–0.22	@ bonds only	0.00000
250	98.3	11.95	0.034	2335/2321	0.041	0.43/–0.25	@ bonds only	0.00000
300	98.1	9.77	0.035	2351/2345	0.050	0.50/–0.28	@ bonds only	0.00000
10	98.3	25.59	0.036	1211/1211	0.026	0.28/–0.29	@ bonds only	0.00000
108	98.5	20.69	0.037	1190/1190	0.029	0.35/–0.35	@ bonds only	0.00000
200	97.9	18.85	0.038	1190/1189	0.032	0.30/–0.30	@ bonds only	0.00000
300	98.4	19.75	0.034	1189/1197	0.036	0.40/–0.33	@ bonds & atoms	0.00000

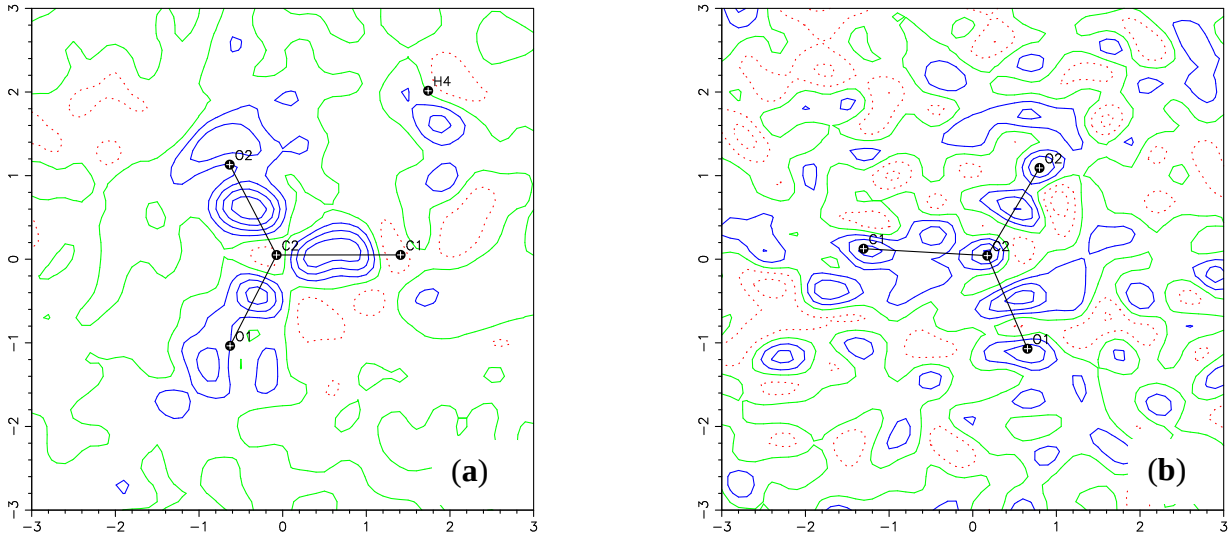

Fig. 1. Residual density maps of (a) α- glycine and (b) γ-glycine data at 300 K from beamline ID11.

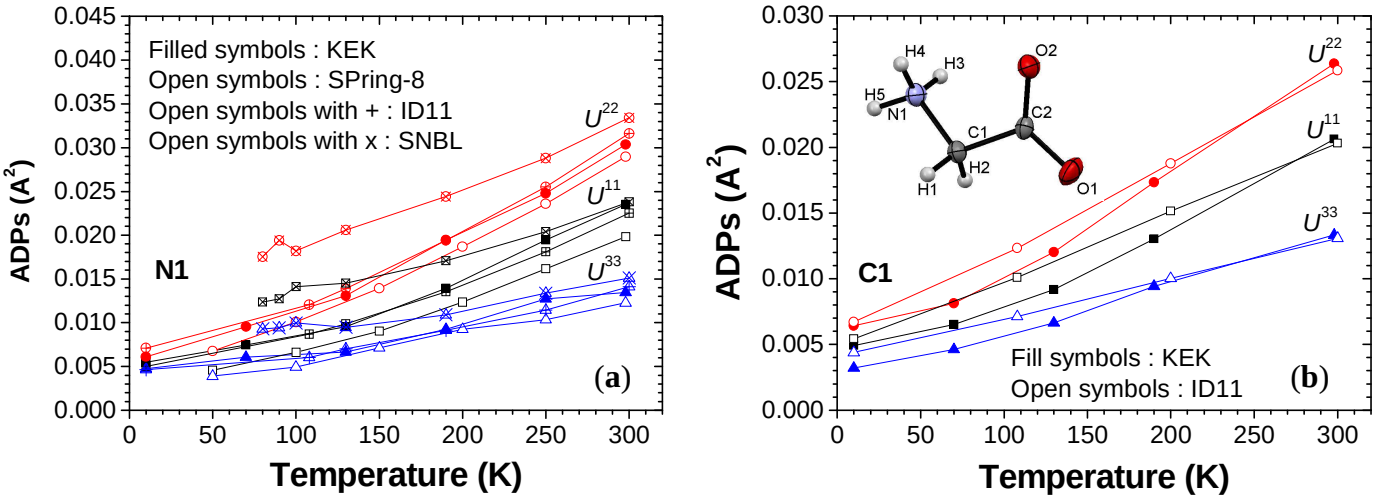

Fig. 2. Variable temperature ADPs for (a) N1 of α-glycine and (b) C1 of γ-glycine from beamline ID11 in comparison with other sources. Standard deviations are 2×10^{-4} Å^2.

References:

[1] Bürgi, H.B.; Capelli, S.C. *Acta Cryst*. **2000**, *A56*, 403–412.

[2] Machida, K.; Kagayama, A.; Saito, Y.; Kuroda, Y.; Uno, T. *Spectrochim. Acta* **1977**, *33A*, 569–574.

[3] Baran, J.; Ratajczak H. *Spectrochim. Acta* **2005**, *61A*, 1611–1626.