

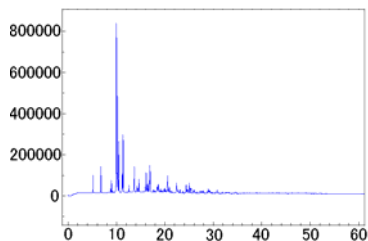
粉末回折における新しい 未知構造決定手法の提案

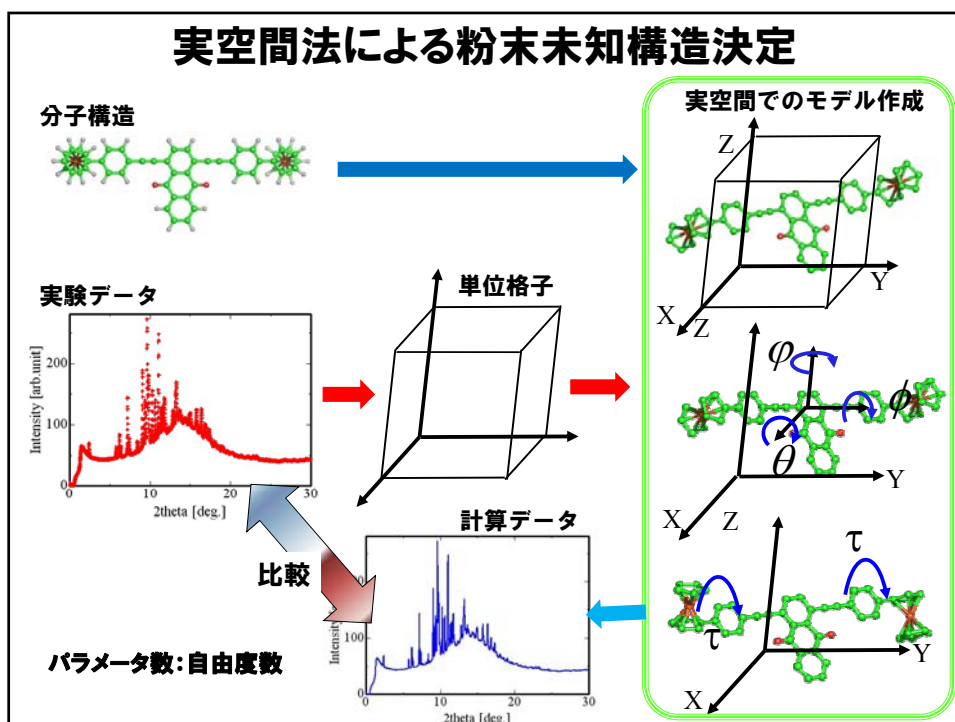
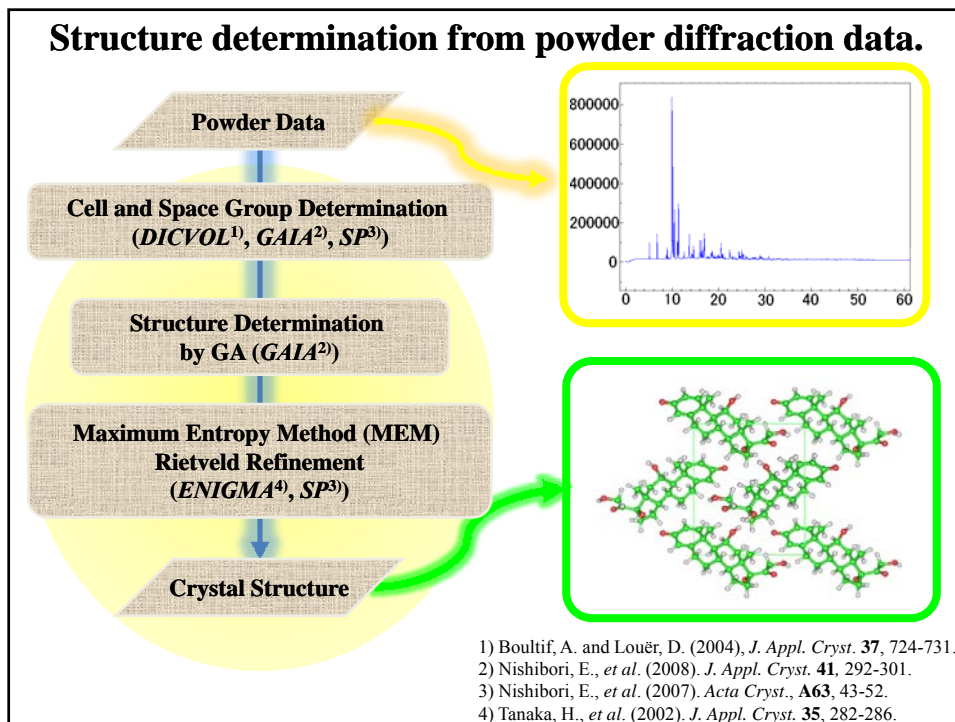
名古屋大学大学院工学研究科
マテリアル理工学専攻応用物理学分野
西堀 英治

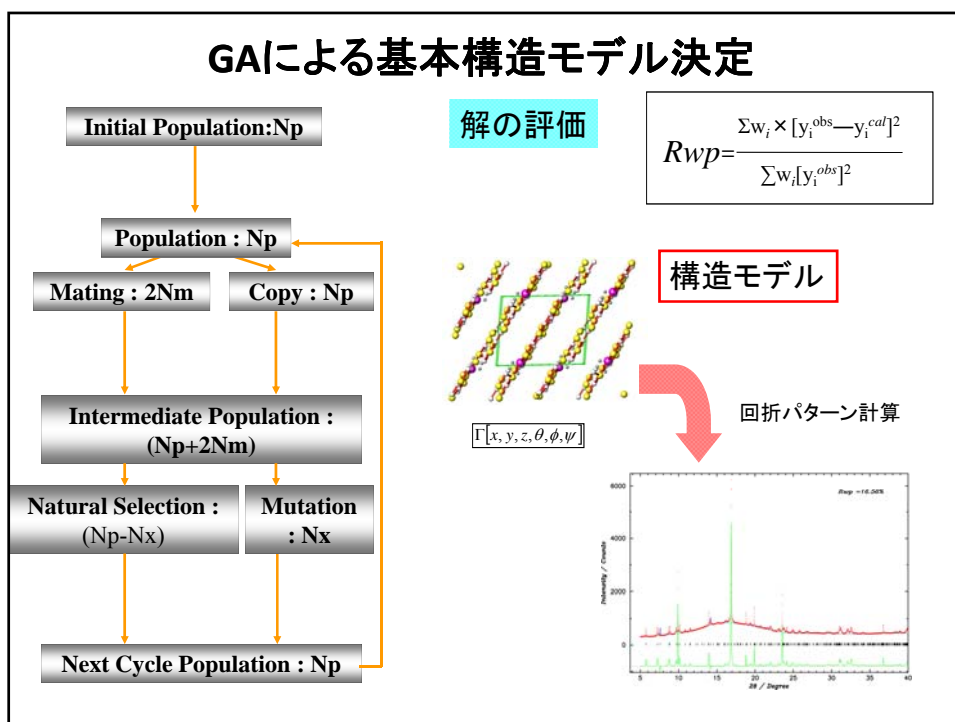
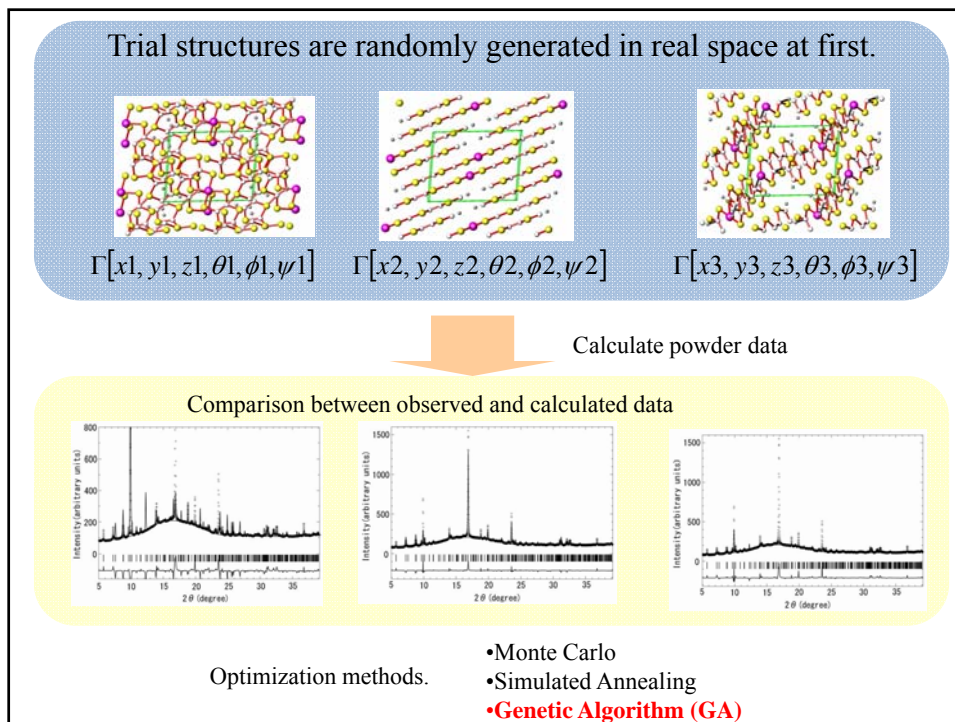
High Resolution Powder diffraction data
by advanced quantum beam source

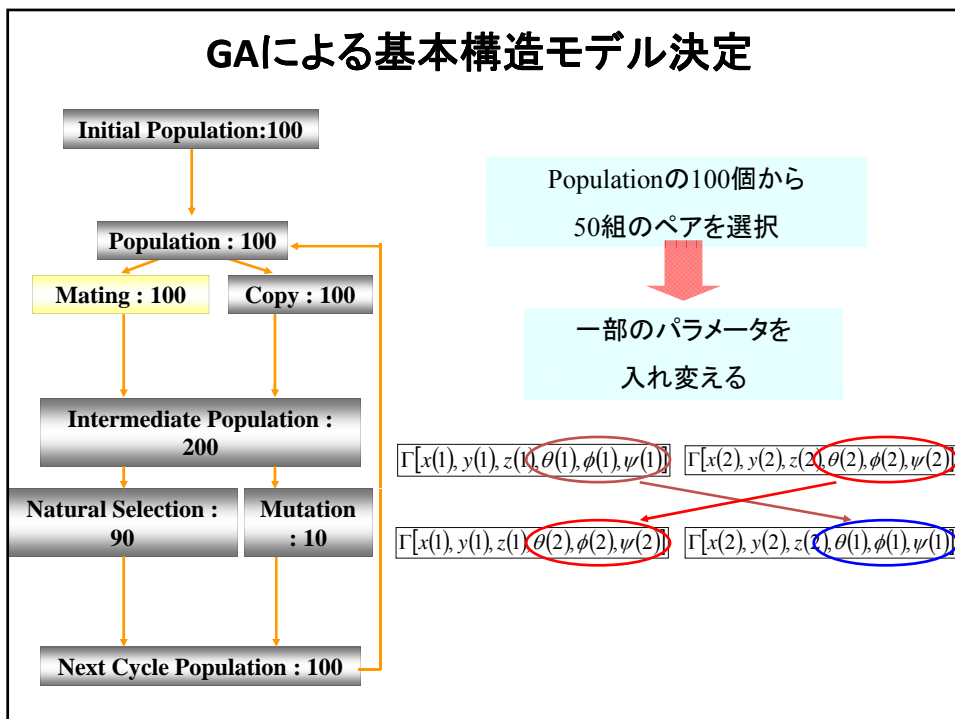
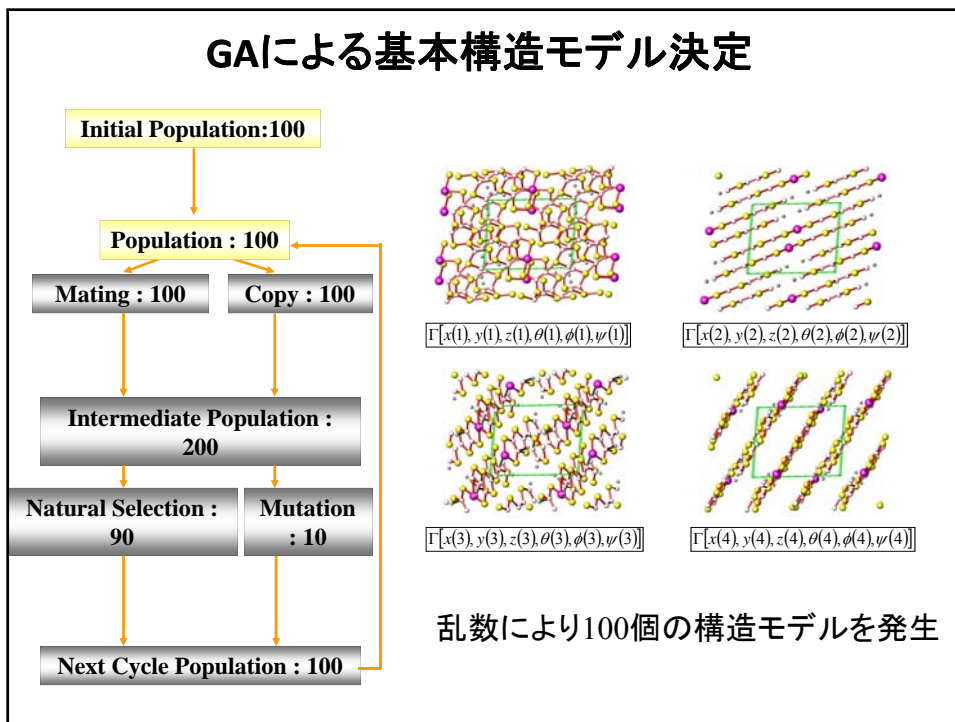


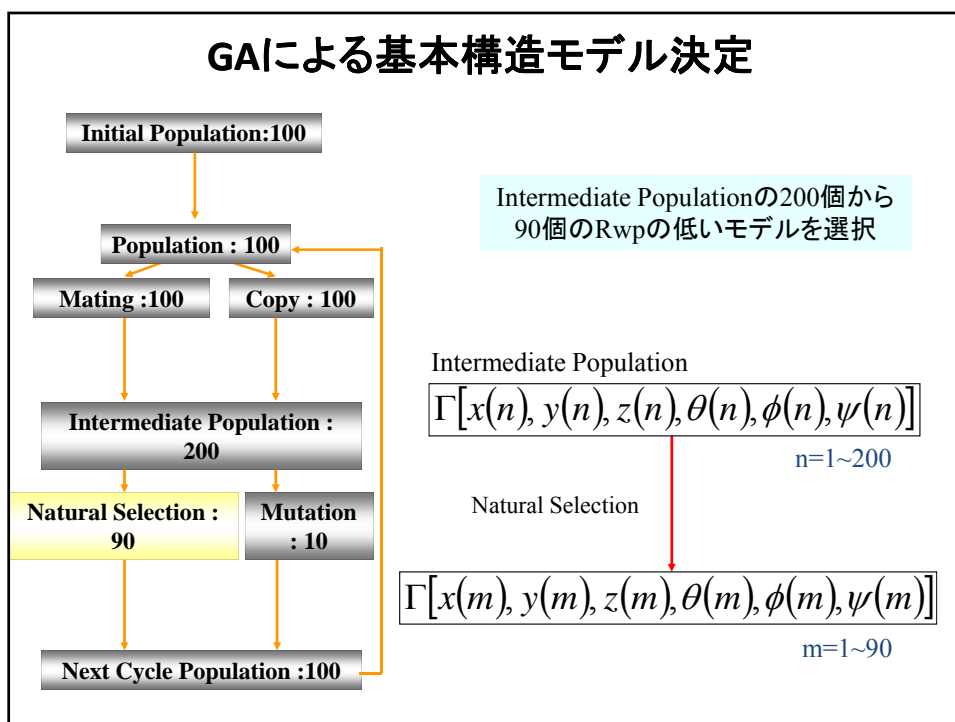
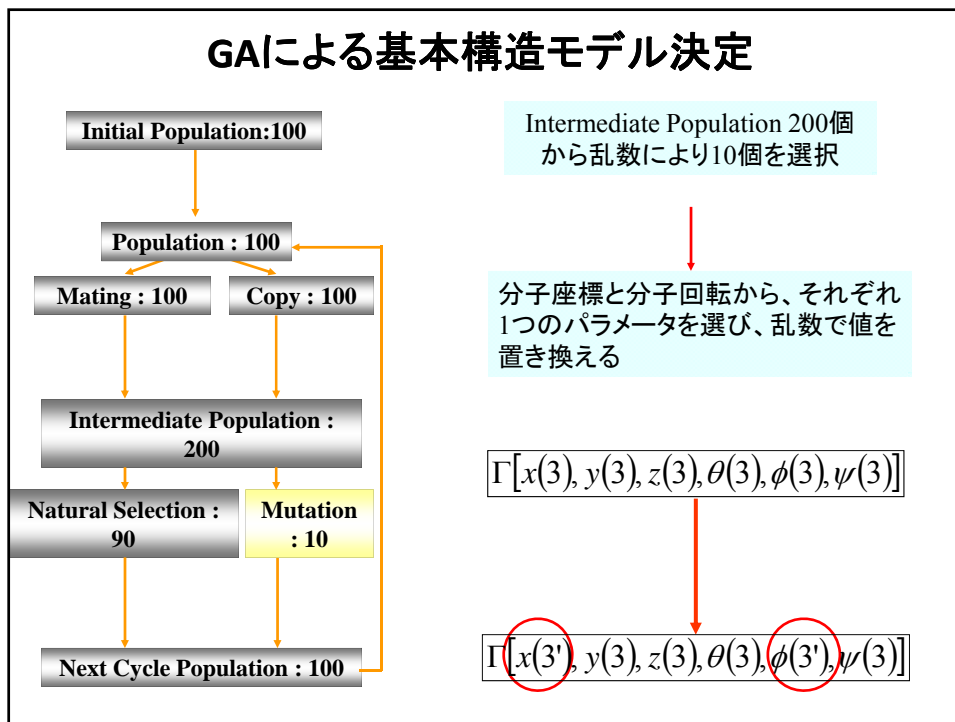
High Quality Diffraction Data







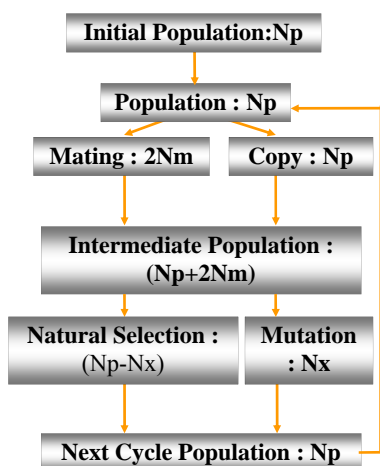




遺伝的アルゴリズムを用いた構造決定

11

GA for Structure Determination (K. D. M. Harris et. al.,)



単成分分子からなる 分子性金属

- Ni(dt)₂, Pd(dt)₂
- Co(dt)₂
- Zn(tmdt)₂, Au(tmdt)₂

有機伝導体関連物質 D-A共役接合錯体

- 1-4-Fc₂Bz₂Aq

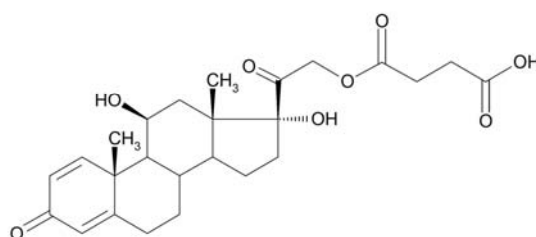
医薬品

- プレドニゾロン

6~15自由度を持つ物質の構造決定

坂田 誠、西堀 英治 特願 2005-350770,
PCT/JP2006/324614

Structure determination of Pharmaceutical



Prednisolone Succinate
(11b,17-dihydroxypregna-1,4-diene-3,20-dione-21-succinate)

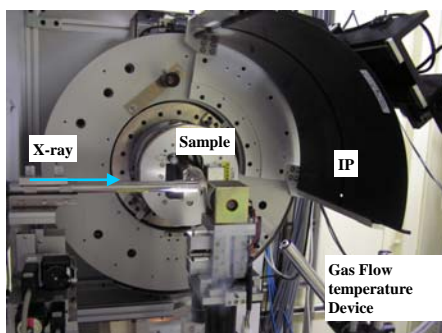
Steroid drug

molecular formula: C₂₅H₃₂O₈

molar weight: 460.52

Powder Diffraction Experiment

Spring-8 BL02B2

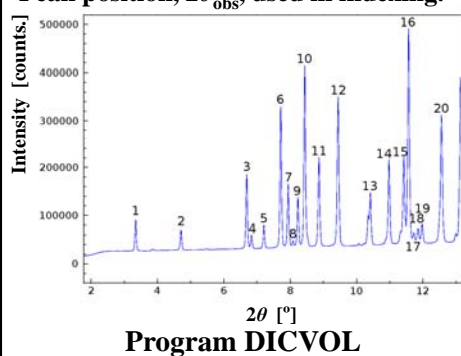


Sample sealed in 0.4mm ϕ capillary
 Wave Length $\lambda = 1.0 \text{ \AA}$
 Range: $2\theta = 2 \sim 75^\circ$ ($d > 0.82 \text{ \AA}$)
 Exposure Time 145 min.
 Temperature 100, 300, 400 K



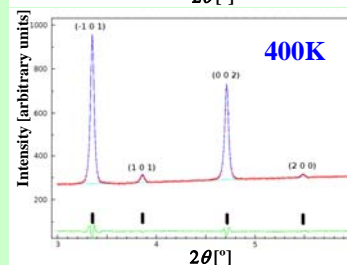
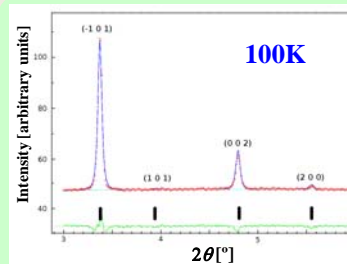
Cell & Space Group Determination: Indexing.

Peak position, $2\theta_{\text{obs}}$, used in indexing.

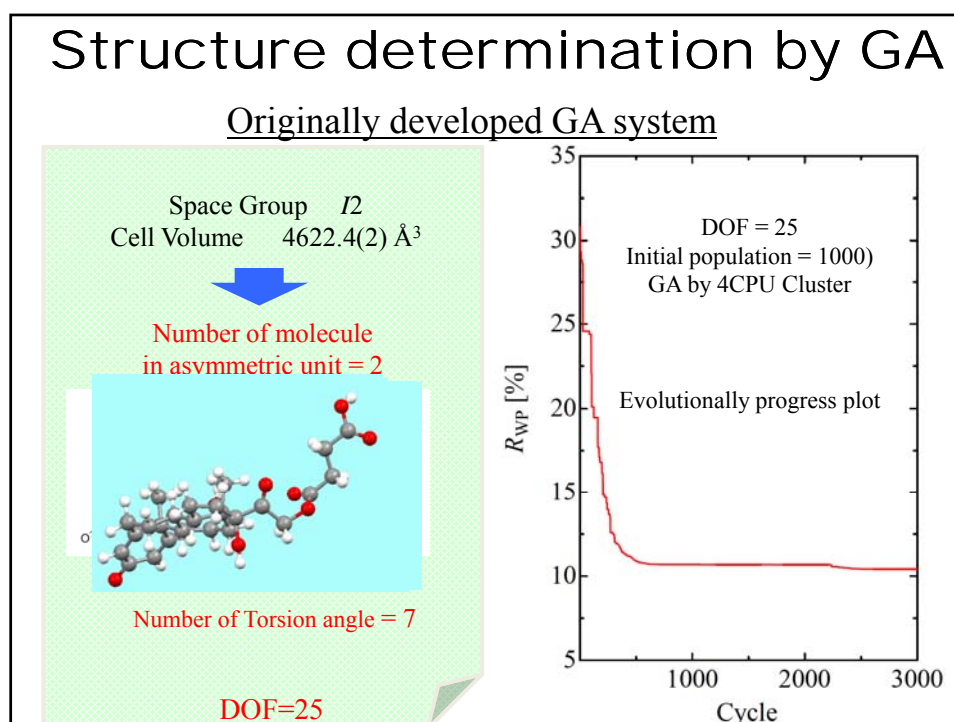
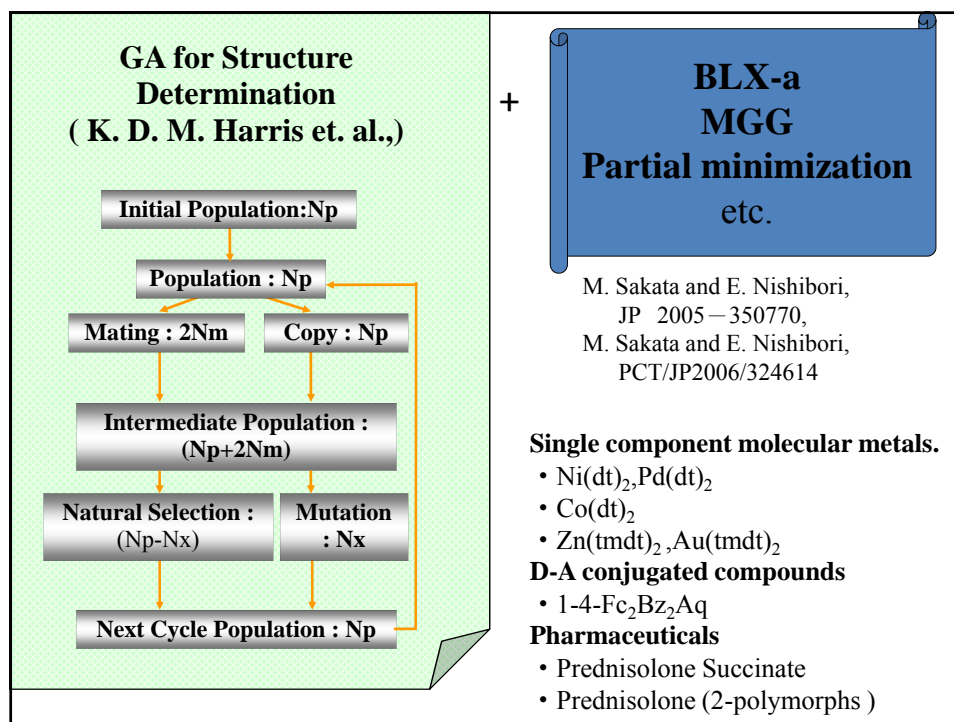


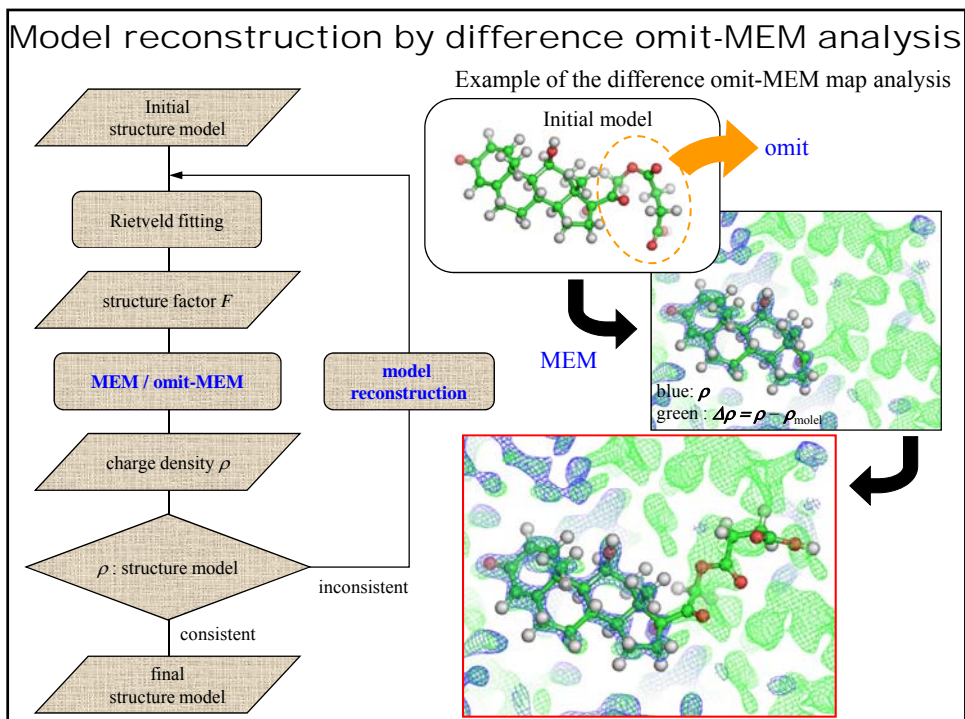
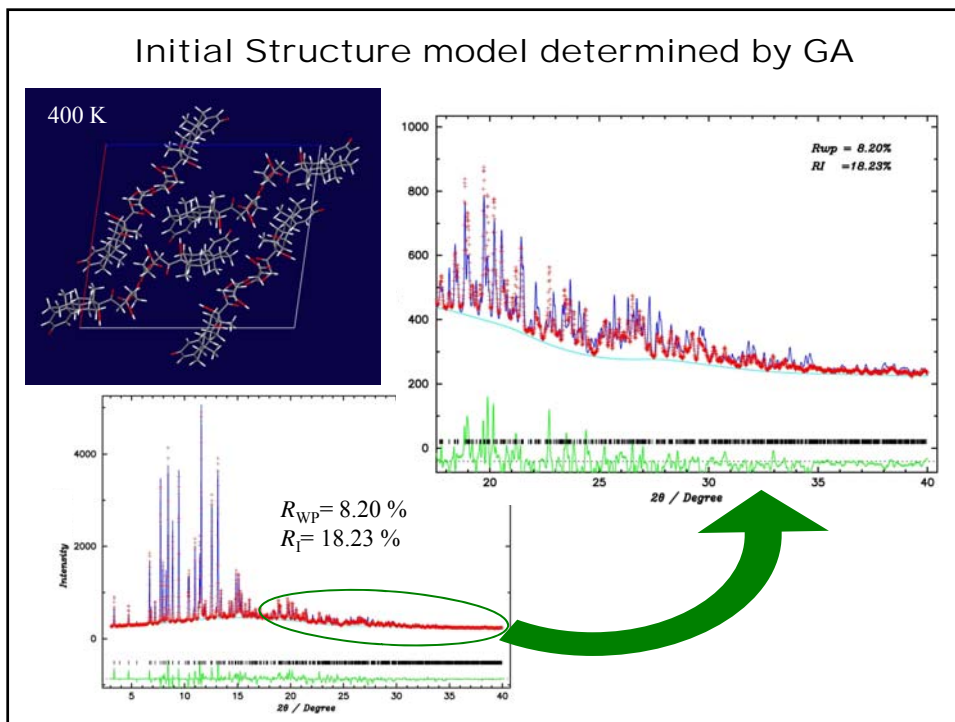
Lattice Constants refined by Le Bail method.

Monoclinic :
 $a = 21.13906 \text{ [\AA]}$, $b = 9.16104 \text{ [\AA]}$, $c = 24.58806 \text{ [\AA]}$,
 $\beta = 98.1434 \text{ [}^\circ\text{]}$



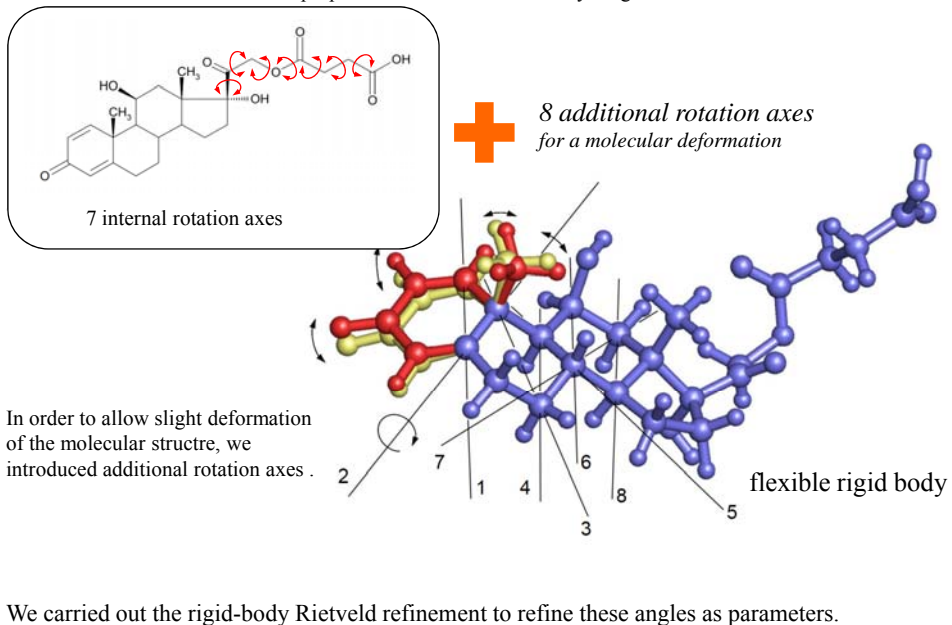
Multi-temperature data were used for confirmation.





Rietveld refinement using flexible rigid body.

It is still difficult to obtain a proper solution without relatively longer and shorter bond distances.



Final result of Refinements

