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**Supporting information for article:**

# The *Shift_and_Fix* procedure in *EXPO*: advances for solving *ab initio* crystal structure by powder diffraction data

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**Table S1** For each test structure: compound name and code name, molecular formula and reference.

| | **compound name**<br>**code name** | **molecular formula** | **Reference** |
|---|---|---|---|
| **1.** | Methylsodium<br>METYL | $NaCH_3$ | Weiss *et al.* (1990) |
| **2.** | Copper(I) pyrazolate<br>CUPZ | $C_3H_3CuN_2$ | Masciocchi *et al.* (1994) |
| **3.** | N-formylu<br>FORMYLU | $C_2H_4N_2O_2$ | Harris *et al.* (1998) |
| **4.** | Potassium uranyl phosphate trihydrate,<br>KUOS | $KUO_2PO_4.3H_2O$ | Fitch & Cole (1991) |
| **5.** | *N,N'*-Bis[1-pyridin-4-yl-meth-(*E*)-ylidene]hydrazine<br>HYDRAZINE | $C_{12}H_{10}N_4$ | Florence *et al.* (2005) |
| **6.** | Benzene-Hexafluorobenzene<br>BENZ | $C_6H_6$:$C_6F_6$ | Williams *et al.* (1992) |
| **7.** | Magnesium perchlorate hydrate<br>PCHLORO | $Mg(ClO_4)_2 \cdot 4H_2O$ | Robertson & Bish (2010) |
| **8.** | $\alpha$ phase of L-glutamic acid<br>ALPHA | $C_5H_9NO_4$ | Kariuki *et al.* (1998) |
| **9.** | Creatine monohydrate<br>CREATINE | $C_4H_9N_3O_2 \cdot H_2O$ | Florence *et al.* (2005) |
| **10.** | 2-Mercaptobenzoic acid<br>MERCA | $C_7H_6O_2S$ | Florence *et al.* (2005) |
| **11.** | Phenylacetic acid<br>PHENYLA | $C_8H_8O_2$ | Florence *et al.* (2005) |
| **12.** | Paracetamol (form I polymorph)<br>PARACETAMOL1 | $C_8H_9NO_2$ | Florence *et al.* (2005) |
| **13.** | RS-camphorquinone<br>CAMPHOR | $C_{10}H_{14}O_2$ | unpubl. data by courtesy of Dr. M. Brunelli, Grenoble, France |
| **14.** | *cis*-inositol (5-A)<br>INOSITOL5A | $C_6H_{12}O_6$ | Beko *et al.* (2014) |
| **15.** | *cis*-inositol (5-D)<br>INOSITOL5D | $C_6H_{12}O_6$ | Beko *et al.* (2014) |
| **16.** | D-(+)-chiro-inositol (D-1-A)<br>INOSITOLD1A | $C_6H_{12}O_6$ | Beko *et al.* (2014) |
| **17.** | Aspirin<br>ASPIRINS | $C_9H_8O_4$ | diffraction data by courtesy of Professor N. Masciocchi, |

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|---|---|---|---|
| | | | Como, Italy (http://en.wikipedia.org/wiki/Aspirin) |
| **18.** | 2-(Benzoylsulfanyl)acetic acid BENZOS1 | $C_9H_8O_3S$ | Rukiah & Al-Ktaifani (2011) |
| **19.** | Carbamazepine dihydrate CARBA | $C_{15}H_{12}N_2O{\cdot}2H_2O$ | Florence *et al.* (2005) |
| **20.** | Citric acid CITRIC_ACIDD8 | $C_6H_8O_7$ | diffraction data by courtesy of Professor N. Masciocchi, Como, Italy (http://en.wikipedia.org/wiki/Citric_acid) |
| **21.** | Citric acid CITRIC_ACIDS | $C_6H_8O_7$ | diffraction data by courtesy of Professor N. Masciocchi, Como, Italy (http://en.wikipedia.org/wiki/Citric_acid) |
| **22.** | 2-[N-morpholino] ethane sulfonic acid hydrate MES | $C_6H_{15}N_1O_5S_1$ | Christensen *et al.* (1993) |
| **23.** | Sulfuric acid octahydrate OCTAH | $H_2SO_4$ $8H_2O$ | Maynard-Casely *et al.* (2012) |
| **24.** | 2-[1-(2-aminoethyl)-2-imidazolidinylidene]-2-nitroacetonitrile AND1 | $C_7H_{11}N_5O_2$ | Chernyshev *et al.* (1999) |
| **25.** | Calcium Glicinate trihydrate CAGLY | $Ca_2C_4H_{14}N_2O_7$ | Le Bail *et al.* (2013) |
| **26.** | Captopril CAPTO | $C_9H_{15}NO_3S$ | Fujinaga & James (1980) |
| **27.** | Lanthanum Oxomolybdate LAMO | $La_1Mo_5O_8$ | Hibble *et al.* (1988) |
| **28.** | 2-(4-Hydroxy-2-oxo-2,3-dihydro-1,3-benzothiazol-7-yl)ethylammonium chloride AMMONIUM | $C_9H_{11}N_2O_2S{\cdot}Cl$ | Florence *et al.* (2005) |
| **29.** | Ibuprofen IBUPS | $C_{13}H_{18}O_2$ | Shankland *et al.* (1998) |

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|---|---|---|---|
| **30.** | Calcium tartrate tetrahydrate<br>TARTRATE | $Ca_1C_4H_4O_6{\cdot}4H_2O$ | Le Bail *et al.* (2009) |
| **31.** | N-(2,6-dimethylphenyl)-5,6-dihydro-4H-1,3-thiaz-2-amine hydro-chloride<br>XYLAZINEA | $C_{12}H_{17}Cl_1N_2S_1$ | Zvirgzdins *et al.* (2014) |
| **32.** | N-(2,6-dimethylphenyl)-5,6-dihydro-4H-1,3-thiaz-2-amine hydro-chloride<br>XYLAZINEZ | $C_{12}H_{17}Cl_1N_2S_1$ | Zvirgzdins *et al.* (2014) |
| **33.** | *catena*-Poly[[dipyridinenickel(II)]-trans-di-μ-chlorido]<br>DIPYR | $[NiCl_2(C_5H_5N)_2]_n$ | Alig *et al.* (2010) |
| **34.** | *m*-Toluidine<br>LEFEBVRE | $C_{56}N_8$ | Rukiah *et al.* (2004) |
| **35.** | 2,6-diamino-5-hydroxy-3-nitro-4*H*-pyrazolo[1,5-a]pyrimidin-7-one monohydrate<br>AND2 | $C_6H_6N_6O_4{\cdot}H_2O$ | Chernyshev *et al.* (1999) |
| **36.** | Cimetidine<br>CIME | $C_{10}H_{16}N_6S$ | Cernik *et al.* (1991) |
| **37.** | Dapsone<br>DAPSONE | $C_{12}H_{12}N_2O_2S$ | Florence *et al.* (2005) |
| **38.** | 1,4-Bis(2-phenethyloxyethanesulfonyl) piperazine<br>PIPERAZINE | $C_{24}H_{34}N_2O_6S_2$ | Florence *et al.* (2005) |
| **39.** | Salbutamol<br>SALBU | $C_{13}H_{21}NO_3$ | Florence *et al.* (2005) |
| **40.** | 4,4′-diiso-cyano-3,3′-di-methyl-biphen-yl<br>DICYANO | $C_{64}N_8$ | Rukiah & Al-Ktaifani(2013) |
| **41.** | Nickel zirconium phosphate<br>NIZR | $Ni_{0.5}Zr_2(PO_4)_3$ | Jouanneaux *et al.* (1991) |
| **42.** | Yttrium nitrate<br>YONO | $Y_4O_{13}N_1H_9$ | Christensen et *al.* (1992) |
| **43.** | Triphenylphosphine<br>PPH3D8 | $P(C_6H_5)_3$ | http://en.wikipedia.org/wiki/Triphenylphosphine |

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|---|---|---|---|
| **44.** | Zeolite UTM-1<br>UTM1 | $Si_{44}O_{88}$ | Plévert *et al.* (1999) |
| **45.** | 2,5-dioxopyrrolidin-1-yl 2-(benzoylsulfanyl)acetate<br>BENZOS2 | $C_{13}H_{11}NO_5S$ | Rukiah & Al-Ktaifani (2011) |
| **46.** | Tetracaine hydrochloride<br>CAINE | $C_{15}H_{25}N_2O_2^+Cl^-$ | Nowell *et al.* (2002) |
| **47.** | 5,10-dihydro-5,10-dioxonaphtho[2,3-b]-1,4-dithiine-2,3-dicarbonitrile<br>DITHIANON2 | $C1_4H_4N_2O_2S_2$ | Halasz *et al.* (2012) |
| **48.** | Aluminophosphate<br>ALPO | $P_{16}Al_{16}O_{88}N_{16}C_{32}$ | Baerlocher *et al.* (2004) |
| **49.** | *trans*-Dichlorobis(triphenylphosphine)nickel(II)<br>NICKEL | $C_{36}H_{30}Cl_2NiP_2$ | Florence *et al.* (2005) |
| **50.** | Aluminophosphate<br>SAPO | $(SiAlP)_{32}O_{64}C_{24}H_{58}N_2$ | Estermann *et al.* (1992) |
| **51.** | S-Bupivacaine Hydrochloride (Form A)<br>BUPIA | $C_{18}H_{28}N_2O{\cdot}HCl$ | Niederwanger *et al.* (2009) |
| **52.** | Capsaicin<br>CAPSA | $C_{18}H_{27}NO_3$ | Florence *et al.* (2005) |
| **53.** | Niobium V oxophosphate<br>NBPO | $Nb_5P_7P_9$ | Zah-Letho *et al.* (1992) |
| **54.** | Clomipramine hydrochloride<br>CLOMIPRA | $C_{19}H_{24}ClN_2{\cdot}Cl$ | Florence *et al.* (2005) |
| **55.** | Benzyloxycarbonil α-aminoisobutyric acid<br>PEP1 | $C_{16}H_{22}N_2O_5$ | Valle *et a.l* (1986) |
| **56.** | 2,4-dihydroxy-benzoic acid–N′-(propan-2-yl-idene)nicotinohydrazide<br>DHBENZO | $C_{16}H_{17}N_3O_5$ | Lapidus *et al.* (2012) |
| **57.** | *cis* Inositol (5E) | $C_6H_{12}O_6$ | Beko *et al.* (2014) |

| | | | |
|---|---|---|---|
| | INOSITOL5E | | |
| **58.** | Ethyl 1', 2', 3', 4', 4a', 5', 6', 7'octahydrodispiro[cyclohexane-1,2' -quinazoline-4' ,1''-cyclohexane]-8'-carbodithioate<br>DISPIRO | $C_{21}H_{34}N_2S_2$ | Ávila *et al.* (2009) |
| **59.** | Aluminophosphate<br>VFI | $Al_{18}P_{18}O_{114}H_{84}$ | McCusker *et al.* (1991) |

**Table S2_TEST1.** The standard ALLTRIALS (**ALLTs**) and **TEST1** procedures are verified. For each test structure:

- *nau* is the number of non-hydrogen atoms to be located in the asymmetric unit;
- NCA is the number of atoms correctly located in the model first ranked by ALLTRIALS;
- $NP_{min}$ is the minimum order number of the position in the ALLTRIALS ranked list corresponding to a correct solution, whose number of atoms correctly located is larger than that one of the first ranked model. If the best model corresponds to the first ranked one or the correct solution is not attained, $NP_{min}$ is equal to one.
- $NCA_p$ is the number of atom positions correctly located of the structure model ranked position $NP_{min}$ (it is given only when $NP_{min}$ is larger than 1).
- NCS is the number of correct solutions among the ALLTRIALS processed trials.
- <NCA> and <NCS> are the corresponding average values.

| code name | | **ALLTs** | | | | **TEST1** | | | |
|---|---|---|---|---|---|---|---|---|---|
| | *nau* | NCA | $NP_{min}$ | $NCA_p$ | NCS | NCA | $NP_{min}$ | $NCA_p$ | NCS |
| KUOS | 7 | 1 | 3 | 6 | 4 | 0 | 2 | 6 | 2 |
| PCHLORO | 9 | 7 | 2 | 9 | 9 | 9 | 1 | | 9 |
| ALPHA | 10 | 5 | 1 | - | 0 | 4 | 1 | - | 0 |
| PHENYLA | 10 | 3 | 1 | - | 0 | **10** | **1** | **-** | **4** |
| INOSITOL5A | 12 | 2 | 1 | - | 0 | **12** | **1** | **-** | **2** |
| INOSITOLD1A | 12 | 4 | 1 | - | 0 | **12** | **1** | **-** | **1** |
| BENZOS1 | 13 | 12 | 4 | 13 | 10 | 13 | 1 | - | 12 |
| CARBA | 13 | 11 | 1 | - | 2 | *6* | *1* | - | *0* |
| LAMO | 14 | 0 | 4 | 11 | 1 | 14 | 1 | - | 1 |
| IBUPS | 15 | 2 | 1 | - | 0 | 11 | 1 | - | 0 |
| TARTRATE | 15 | 4 | 1 | - | 0 | 5 | 1 | - | 0 |
| XYLAZINEA | 16 | 4 | 6 | 15 | 3 | 7 | 2 | 14 | 2 |
| XYLAZINEZ | 16 | 12 | 2 | 16 | 4 | 16 | 1 | - | 5 |
| DIPYR | 16 | 2 | 1 | - | 0 | 0 | 1 | - | 0 |
| LEFEBVRE | 16 | 1 | 1 | - | 0 | 0 | 1 | - | 0 |
| PIPERAZINE | 17 | 2 | 1 | - | 0 | **17** | **1** | **-** | **1** |
| DICYANO | 18 | 7 | 1 | - | 0 | 13 | 1 | - | 0 |
| NIZR | 18 | 10 | 2 | 15 | 1 | 14 | 1 | - | 3 |
| YONO | 18 | 14 | 2 | 15 | 2 | *11* | *1* | - | *0* |
| PPH3D8 | 19 | 10 | 2 | 18 | 1 | *1* | *1* | - | *0* |
| UTM1 | 19 | 17 | 2 | 18 | 2 | 19 | 1 | - | 2 |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| BENZOS2 | 20 | 2 | 1 | - | 0 | **20** | **1** | **-** | **1** |
| DITHIANON2 | 20 | 6 | 1 | - | 0 | 0 | 1 | - | 0 |
| ALPO | 21 | 20 | 1 | - | 3 | 16 | 2 | 18 | 3 |
| NICKEL | 21 | 20 | 1 | - | 1 | 21 | 1 | - | 4 |
| SAPO | 21 | 9 | 1 | - | 0 | **18** | **1** | **-** | **2** |
| CAPSA | 22 | 0 | 1 | - | 0 | **20** | **1** | **-** | **1** |
| NBPO | 22 | 20 | 1 | - | 1 | 22 | 1 | - | 2 |
| CLOMIPRA | 23 | 18 | 1 | - | 1 | 18 | 1 | - | 1 |
| DHBENZO | 24 | 23 | 1 | - | 1 | 23 | 2 | 24 | 5 |
| INOSITOL5E | 24 | 1 | 8 | 20 | 1 | *0* | *1* | - | *0* |
| VFI | 25 | 21 | 1 | - | 5 | 19 | 2 | 21 | 3 |
| | | <NCA> 8.4 | | | <NCS> 1.6 | <NCA> 11.6 | | | <NCS> 2.1 |

**Table S3_TEST1.** The standard ALLTRIALS (**ALLTs**) and **TEST1** procedures are verified for those structures for which the complete and correct solution (all the non-hydrogen atoms in the asymmetric unit have been correctly located) is attained by both ALLTs and TEST1 and is first ranked by the $R_F$ value. For each test structure:

- *nau* is the number of non-hydrogen atoms to be located in the asymmetric unit;
- NCS is the number of correct solutions among the ALLTRIALS processed trials;
- <NCS> is the corresponding average value.

| code name | | **ALLTs** | **TEST1** |
|---|---|---|---|
| | *nau* | NCS | NCS |
| METYL | 5 | 9 | 11 |
| CUPZ | 6 | 2 | 10 |
| FORMYLU | 6 | 7 | 17 |
| HYDRAZINE | 8 | 2 | 5 |
| BENZ | 9 | 3 | 6 |
| CREATINE | 10 | 4 | 10 |
| MERCA | 10 | 2 | 7 |
| PARACETAMOL1 | 11 | 4 | 12 |
| CAMPHOR | 12 | 1 | 5 |
| INOSITOL5D | 12 | 5 | 8 |
| ASPIRINS | 13 | 5 | 6 |
| CITRIC_ACIDD8 | 13 | 13 | 14 |

| | | | |
|---|---|---|---|
| CITRIC_ACIDS | 13 | 4 | 7 |
| MES | 13 | 8 | 13 |
| OCTAH | 13 | 4 | 5 |
| AND1 | 14 | 2 | 5 |
| CAGLY | 14 | 1 | 1 |
| CAPTO | 14 | 7 | *2* |
| AMMONIUM | 15 | 4 | 8 |
| AND2 | 17 | 3 | 3 |
| CIME | 17 | 1 | 5 |
| DAPSONE | 17 | 4 | *3* |
| SALBU | 17 | 1 | 4 |
| CAINE | 20 | 2 | 5 |
| BUPIA | 22 | 2 | *1* |
| PEP1 | 23 | 2 | 4 |
| DISPIRO | 25 | 8 | *7* |
| | | <NCS> 4.1 | <NCS> 6.8 |

**Table S2_TEST2.** The standard ALLTRIALS (**ALLTs**) and **TEST2** procedures are verified. For each test structure:

- *nau* is the number of non-hydrogen atoms to be located in the asymmetric unit;
- NCA is the number of atoms correctly located in the model first ranked by ALLTRIALS;
- $NP_{min}$ is the minimum order number of the position in the ALLTRIALS ranked list corresponding to a correct solution, whose number of atoms correctly located is larger than that one of the first ranked model. If the best model corresponds to the first ranked one or the correct solution is not attained, $NP_{min}$ is equal to one.
- $NCA_p$ is the number of atom positions correctly located of the structure model ranked position $NP_{min}$ (it is given only when $NP_{min}$ is larger than 1).
- NCS is the number of correct solutions among the ALLTRIALS processed trials.
- <NCA> and <NCS> are the corresponding average values.

| code name | | **ALLTs** | | | | **TEST2** | | | |
|---|---|---|---|---|---|---|---|---|---|
| | *nau* | NCA | $NP_{min}$ | $NCA_p$ | NCS | NCA | $NP_{min}$ | $NCA_p$ | NCS |
| KUOS | 7 | 1 | 3 | 6 | 4 | 5 | 4 | 6 | 4 |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| PCHLORO | 9 | 7 | 2 | 9 | 9 | 8 | 5 | 9 | 8 |
| ALPHA | 10 | 5 | 1 | - | 0 | 4 | 1 | - | 0 |
| PHENYLA | 10 | 3 | 1 | - | 0 | 1 | 1 | - | 0 |
| INOSITOL5A | 12 | 2 | 1 | - | 0 | **12** | **1** | **-** | **2** |
| INOSITOLD1A | 12 | 4 | 1 | - | 0 | **12** | **1** | **-** | **1** |
| BENZOS1 | 13 | 12 | 4 | 13 | 10 | 13 | 1 | | 11 |
| CARBA | 13 | 11 | 1 | - | 2 | 11 | 1 | - | 1 |
| LAMO | 14 | 0 | 4 | 11 | 1 | 13 | 1 | - | 2 |
| IBUPS | 15 | 2 | 1 | - | 0 | 3 | 1 | - | 0 |
| TARTRATE | 15 | 4 | 1 | - | 0 | 1 | 1 | - | 0 |
| XYLAZINEA | 16 | 4 | 6 | 15 | 3 | 11 | 6 | 14 | 3 |
| XYLAZINEZ | 16 | 12 | 2 | 16 | 4 | 16 | 1 | - | 2 |
| DIPYR | 16 | 2 | 1 | - | 0 | 11 | 1 | - | 0 |
| LEFEBVRE | 16 | 1 | 1 | - | 0 | 5 | 1 | - | 0 |
| PIPERAZINE | 17 | 2 | 1 | - | 0 | 0 | 1 | - | 0 |
| DICYANO | 18 | 7 | 1 | - | 0 | 5 | 1 | - | 0 |
| NIZR | 18 | 10 | 2 | 15 | 1 | *8* | *1* | - | *0* |
| YONO | 18 | 14 | 2 | 15 | 2 | 16 | 1 | - | 2 |
| PPH3D8 | 19 | 10 | 2 | 18 | 1 | 19 | 1 | - | 1 |
| UTM1 | 19 | 17 | 2 | 18 | 2 | 19 | 1 | - | 4 |
| BENZOS2 | 20 | 2 | 1 | - | 0 | 0 | 1 | - | 0 |
| DITHIANON2 | 20 | 6 | 1 | - | 0 | 5 | 1 | - | 0 |
| ALPO | 21 | 20 | 1 | - | 3 | 18 | 2 | 20 | 4 |
| NICKEL | 21 | 20 | 1 | - | 1 | 21 | 1 | - | 2 |
| SAPO | 21 | 9 | 1 | - | 0 | 12 | 1 | - | 0 |
| CAPSA | 22 | 0 | 1 | - | 0 | 0 | 1 | - | 0 |
| NBPO | 22 | 20 | 1 | - | 1 | 21 | 1 | - | 2 |
| CLOMIPRA | 23 | 18 | 1 | - | 1 | *3* | *1* | - | *0* |
| DHBENZO | 24 | 23 | 1 | - | 1 | 0 | 2 | 23 | 1 |
| INOSITOL5E | 24 | 1 | 8 | 20 | 1 | 4 | 17 | 22 | 1 |
| VFI | 25 | 21 | 1 | - | 5 | 19 | 2 | 22 | 5 |
| | | <NCA> 8.4 | | | <NCS> 1.6 | <NCA> 9.3 | | | <NCS> 1.8 |

**Table S3_TEST2.** The standard ALLTRIALS (**ALLTs**) and **TEST2** procedures are verified for those structures for which the complete and correct solution (all the non-hydrogen atoms in the asymmetric unit have

been correctly located) is attained by both ALLTs and TEST2 and is first ranked by the $R_F$ value. For each test structure:

- *nau* is the number of non-hydrogen atoms to be located in the asymmetric unit;
- NCS is the number of correct solutions among the ALLTRIALS processed trials;
- <NCS> is the corresponding average value.

| | | **ALLTs** | **TEST2** |
|---|---|---|---|
| code name | *nau* | NCS | NCS |
| METYL | 5 | 9 | 9 |
| CUPZ | 6 | 2 | 5 |
| FORMYLU | 6 | 7 | 9 |
| HYDRAZINE | 8 | 2 | 5 |
| BENZ | 9 | 3 | 3 |
| CREATINE | 10 | 4 | 6 |
| MERCA | 10 | 2 | 3 |
| PARACETAMOL1 | 11 | 4 | 4 |
| CAMPHOR | 12 | 1 | 2 |
| INOSITOL5D | 12 | 5 | 5 |
| ASPIRINS | 13 | 5 | 7 |
| CITRIC_ACIDD8 | 13 | 13 | 15 |
| CITRIC_ACIDS | 13 | 4 | 4 |
| MES | 13 | 8 | 10 |
| OCTAH | 13 | 4 | *3* |
| AND1 | 14 | 2 | 4 |
| CAGLY | 14 | 1 | 2 |
| CAPTO | 14 | 7 | *5* |
| AMMONIUM | 15 | 4 | 4 |
| AND2 | 17 | 3 | 3 |
| CIME | 17 | 1 | 2 |
| DAPSONE | 17 | 4 | *1* |
| SALBU | 17 | 1 | 1 |
| CAINE | 20 | 2 | *1* |
| BUPIA | 22 | 2 | *1* |
| PEP1 | 23 | 2 | 2 |
| DISPIRO | 25 | 8 | 8 |

| | | | |
|---|---|---|---|
| | | <NCS> 4.1 | <NCS> 4.6 |

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