Supplementary Material

Economy of Catalyst Synthesis—Convenient Access to Libraries of Di- and Tetranaphtho Azepinium Compounds

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Large Scale Synthesis of Non-Racemic 1,1’-Binaphthyl-2,2’-dicarboxylic acid 6 and Sequence Products 7 and 8. (Comparison of methods reported in the literature)

The discussion of preparative scale synthesis yielding enantiopure dihydroazepine 8 via diacid 6 from commercially available starting material will focus on practical aspects (Scheme S1 and Table S1).

The synthesis of enantiomers of 8 requires twelve steps when starting from 2-methylnaphthalene (20) and might include one optical resolution procedure (Scheme S1). With this sequence a total yield of 30% can be expected based on substrate quantities reported in the literature (Table S1). In early steps the reactions were run on typically 40-400 mmol scale with the exception of step d where a 12.7 mmol scale was reported. For the late steps h-k up to 15 mmol of substrate could be reacted with usual laboratory equipment without problems.

Enantiomers: Non-racemic material was commonly obtained by classical optical resolution of diastereomeric compounds / salts at the stage of the diacid 6. Three practicable methods g1-g3 should be considered. The use of brucine as a resolving agent (g1) is hampered due to high price and toxicity. Method g2 uses the less expensive non-racemic 1-phenylethylamine but requires additional steps to
cleave diastereomeric amides. In method g3, finally, the preferred formed 1:2 salt crystallizes and was separated from the mother liquor. The resolving agent was recovered in good yield but following this protocol only one enantiomer of 6 was obtained. The yields of all methods are typically in a range of 40% for each enantiomer.

An interesting report was published which significantly shortens the synthesis. The oxidation of 1-bromo-2-methylnaphthalene (21) with \( \text{O}_2 \) catalysed by \( \text{Co(OAc)}_2 \) giving 24 (step m) is conducted in a steel autoclave and substitutes three steps b-d. The apparently easy operation without purification and good yield (87%) on a large scale (482 mmol) makes this protocol very attractive saving time and manpower (same overall yield as b-d within 1%). Merely, the requirement of a 1L-autoclave which might be not generally available is unfavourable.

Scheme S2: Synthesis of 6 from 29 (Route B)

An alternative route to non-racemic 6 starts from (R)- or (S)-2,2'-dihydroxy-1,1'-binaphthyl (29) which can be obtained by optical resolution using fractional crystallisation of \( N \)-benzylcinchonidinium
clathrate complexes on a 100 g scale but is also commercially available at a reasonable price. For the preparation of 2,2'-dimethyl-1,1'-binaphthyl (31) a Kumada coupling of bistriflate 30 with MeMgCl, MeMgBr or MeMgl and Ni(dppp)Cl₂ as catalysts worked well. The reaction proceeded on a 10-23 mmol scale without racemisation and was frequently reported. Both, 30 and 31 were isolated in pure form after simple filtration over silica in >99% and 95-99% yield, respectively.

Dimethylbinaphthyl 31 was also obtained from 21 as a racemate or enantioselectively using chiral catalysts. The Kumada type biaryl coupling was performed with aryl-Grignard reagents and aryl bromides catalysed by Ni complexes to yield racemic 31 (61%, 12.6 mmol scale, 1 mol% Ni(PPh₃)₂Cl₂) or enantioenriched 31 (69%, 13 mmol scale, 1 mol% (S)(R)-PPFOMe, 95% e.e. (R)-configuration). Other protocols (including Suzuki-Miyaura coupling) requiring expensive chiral catalysts and/or starting materials, seem less appropriate for multigram preparation.

For the stepwise oxidation of 31 to 6 NBS bromination was applied followed by hydrolysis/oxidation either via diol 34 or directly from 32 to dialdehyde 33 which was finally treated with KMnO₄ in acetone/water or H₂O₂, NaClO₂, NaH₂PO₄ in MeCN/water to afford 6. Yields are good to fair (n-o-p-q-s-t: 63% overall yield or v-p-r-s-t: 47% overall yield). When comparing Route A with Route B the latter one will be preferable if non-racemic 6 is desired and enantiopure binaphthol 29 is available. Disadvantageous is the need of expensive triflic anhydride in step m.

Finally, a two step sequences from 29 to 6 might be considered as well. In an early report the bistriflate 30 was methoxycarbonylated under Pd(II)/dppp catalysis to afford the dimethylester of 6 in 83% yield. The use of CO and requirement of noble metal catalysis obviously hampered upscaling and broad use of this protocol. Two other processes working on gram scale were recently reported. After transformation of 29 to 2,2'-diethylphosphate 35 (quant. yield) this was treated with Li-naphthalenide at -78 °C to give the di-lithio compound which reacted with CO₂ to afford 6 in up to 89% yield (3.6 mmol scale). The need of a column chromatography to purify 6 makes up-scaling more difficult (5 mmol scale reported). In an other report triflate 30 was converted to diphenylester 36 using phenylformiate as CO source and Pd(OAc)₂/DPPP as catalyst which was followed by hydrolysis to afford 6. It is worth noting that both processes can be performed stereoconservative, i.e. without racemisation.

Summarizing, for multigram synthesis of 1,1'-binaphthyl-2,2'-dicarboxylic acid (6) two comparable routes are available, starting from either 2-methylnaphthalene (20) (Route A, Scheme S1) or 2,2'-dihydroxy-1,1'-binaphthyl (29) (route B, Scheme S2). Preference will be given depending on the need of racemic or non-racemic material. In the first case Route A is more convenient and requires 4 steps (if reaction n can be performed) or 6 steps with an overall yield of 66-67%. If at this stage an optical resolution is performed the yield will drop to 25-26% for each enantiomer of 6. In this case Route B is superior yielding 63% of non-racemic 6. In contrast, the asymmetric biaryl coupling (Scheme S2, v) requiring expensive catalysts and long reaction time is less appropriate particularly for large scale
preparations. An evaluation of both routes based on time and manpower requirement is rather difficult as the reported time for each step in Table S1 is a rough estimate on the published procedures and do not include preparation/drying/evaporation of solvents. Nevertheless, for the preparation of 5-10 g of 6 an approximate time frame with 10-12 days for Route A and 7-9 days more for optical resolution (g2), and 10-11 days for route B will be a valid approximation.

Comments on Table S1

a: While the bromination of 2-methylnaphthalene (20) with Br₂ in CS₂ yields up to 91% of 21 after distillation, we found the use of HBr/H₂O₂ a more convenient method which could be upscaled to 0.5 mol yielding 95% of the desired product without purification (> 98%, NMR) and sufficiently pure for the next step.

b: Treatment with excess NBS / AIBN in benzene or CCl₄ gave the tribromide 22 in excellent yield.

c: The conversion to aldehyde 23 proceeds smoothly and should also work on multigram scale.

d: Although KMnO₄ oxidation performs satisfying the absence of heavy metal residues with the system NaClO₂/KH₂PO₄ makes it more appropriate.

e: For esterification of 24 several protocols can be applied. Due to price and toxicity of MeI e2 is limited to small scale preparations. The cheapest one is obviously the combination SOCl₂/MeOH. No chromatography is needed.

f: Many binaphthyl coupling methods are known but from the practical point of view the classical Ullmann coupling in DMF is still attractive due to simplicity of the procedure, easy work-up and good yields. Copper powder was activated by treatment with EDTA solution. The crude dimethyl 1,1'-binaphthalene-2,2'-dicarboxylate was immediately hydrolysed and after extractive purification is sufficiently pure.

g: At this stage an optical resolution may be performed.

h-k: These steps were already published for enantiomerically pure substrates and largely omit chromatographic purification. Only for step k the mother liquor from the crystallisation was chromatographed. Repetition with racemic substrate gave comparable yields (±2%).

i: Reaction with aqueous ammonia yielded exclusively the secondary amine 8, provided the reaction temperature was kept at 60 °C. No tertiary amine or spiro-ammonium compound was detected.
Table S1. Synthesis of Diiodoazepine 8 from 2-Methylnaphthalene 22 (Overview)

| Step | Reagent/conditions | Scale (Mmol) | Purification | Time | Yield | Notes |
|------|-------------------|--------------|--------------|------|-------|-------|
| a22  | HBr/H2O           | 1            | no           | 2 d  | 95%   | a     |
| b23  | NBS/ABIN          | 42.5         | chrom.       | 2 d  | 97%   |       |
| b24  | NBS/ABIN          | 10           | chrom.       | 2 d  | 95%   |       |
| c23  | CaCO3/water       | 41           | cryst.       | 1 d  | 95%   |       |
| c24  | AgOAc/acetone-water | 10        | chrom.       | 2 d  | 95%   |       |
| d25  | NaClO4/KH2PO4     | 12.7         | no           | 1 d  | 94%   |       |
| e26  | H2SO4/MeOH        | 1            | no           | 1 d  | 72%   |       |
| e27  | K2CO3/MeI         | 419          | no           | 8 h  | 96%   |       |
| f1   | Brucine           | 88           | cryst.       | 4-5 d| 40(R)/45(S)% |       |
| g1   | 1. (S)-1-phenethyl-amine, DCC/THF, MeCN | 43.8 | crist. | ~7 d | 39(S)/38(R)% | c,d |
| g2   | 2. SOCl2, MeOH, KOH |          |              |      |       |       |
| g3   | (R)-CHEA,MeNH/MeOH | 30        | crist.       | 2 d  | 38(R)% | c,d |
| h28  | n-BuLi, TMP, Me3SiCl, THF | 15 | precip.     | 2 d  | 84%   | t,g   |
| i28  | BH3/THF           | 15           | no           | 2 d  | 84%   | t,g   |
| j28  | ICI/DCM           | 15           | no           | 1 d  | 90%   | t,g   |
| k28  | PBr3/DCM, THF     | 15           | no           | 1 d  | 78%   | t,g   |
| l    | HBr/H2OAc         | 5            | no           | 4 h  | 96%   | this paper |
| m3   | NH3/CH3CN         |              | precip.      | 2 d  | 80-90% | this paper |
| n29  | TEO, 2,6-dimethylpyridine/DCM | 24      | chrom., crist. | 1 d  | 99%   |       |
| o29  | MeMgBr, DPPP/NiCl3/cyclohexane | 23.6   | chrom., crist. | ~3 d | 90-92% |       |
| p29  | NBS, ABIN, hv/cyclohexane | 14.2   | chrom., crist. | 1 d  | 88%   |       |
| q30  | 1. NaHCO3/DMSO, 2. PDC/DCM | 2.5   | chrom.       | 2 d  | 70%   |       |
| r31  | 1. KOAc, BuNB/DMF, 2. KOH/dioxane-H2O | 8    | chrom.       | 4 d  | 88%   |       |
| s32  | MnO/toluene       | 1.6          | no           | 1 d  | 99%   |       |
| t33  | H2O2, NaClO2, NaH2PO4/H2O, MeCN | 30   | no           | 2 h  | 91%   |       |
12. Ni(PPh₃)₂Cl₂, 21-Mg/benzene/Et₂O  
12.6 distil.  
2 d  
61%  

13. NiBr₂, (S)(R)-PPFOMe, 21-Mg/toluene/Et₂O  
10 chrom.  
5 d  
68%  
95% e.e.  

19. ClP(O)(OEt)₂, NaH/THF  
3.5 chrom.  
4 h  
quant.  

19. Li-naphthalene/THF then CO₂  
3.5 chrom.  
6 h  
89%  

20. phenyl formiate, Pd(OAc)₃/DPPP, iPr₂EtN/neat  
4.0 chrom.  
3 d  
63%  

20. KOH/MeOH, water  
0.4h chrom.  
2 d  
89%  

Legend:  
• Pure by NMR (>98%).  
• Two steps, no purification of intermediate.  
• Optical resolution.  
• Two steps.  
• Only one enantiomer isolated.  
• Yields reported for enantiomerically pure material.  
• Synthesis was conducted on a 15 mmol scale with unchanged yield.  
• Mother liquor was chromatographed.  
• Alternatively purified by crystallisation.  
• After two crystallisations.  
• Excess of Grignard reagent of 21 used.  
• Excess of 21 used; 99% e.e. after one cryst.  
• Reported for enantiopure starting material, no racemisation was observed.  
• In the paper the hydrolysis step is reported only on a 0.4 mmol scale but might be upscaled without problems.
$^1$H- and $^{13}$C-NMR spectra (If not otherwise noted spectra are recorded at room temperature in CDCl₃)
$^{1}A_1 \beta$

\[
\begin{array}{c}
\text{Chemical Structure}
\end{array}
\]
3a
DMSC-d6, 378K

[Chemical structure diagram]

[Shift values: 8.0000, 7.3333, 7.2916, 5.0000, 4.0000, 3.0000, 2.0000, 1.0000, 0.0000]
3b
DMSC-d6, 378K

[Chemical structure image]

[1H NMR spectrum with peaks labeled]
3c
DMSO-d6, 378K

[Chemical Structure Image]

[Graphical Data]

[Chemical Shifts]

[M 61.2115]

[M 57.4833]
4c
solvent: DMSO-d6
378K
$4c$
solvent: DMSO-d$_6$
378K
3,3'-Bis(trimethylsilyl)-[1,1'-biphenyl]-2,2'-dicarboxylic acid
3,3'-Bis(trimethylsilyl)-[1,1'-biphenyl]-2,2'-dicarboxylic acid
(3,3'-Bis(trimethylsilylethyl)-1,1'-biphenyl)-2,2'-dihydridimethanol
(3,3'-Bis(trimethylsilyl)-1,1'-biphenyl)-2,2'-diyl)dimethanol

[Chemical Structure Image]
(3,3'-Diiodo[1,1'-biphenyl]-2,2'-diyl)dimethanol
(3,3'-Diodo-[1,1'-bi(phenyl)-2,2'-diyl])dimethanol
in DMSO-$d_6$
in DMSO-\textit{d}_6
17b

Ar = 2-naphthyl
X-ray Analysis

Experimental data and CCDC-Codes can be found in Table S2. Crystal data, data collection parameters, and structure refinement details are given in Tables S3 to S10. Crystal structures visualized in Figure S1 to S4.

**Table S2: Experimental parameter and CCDC-Code.**

| Sample | Machine       | Source | Temp. | Detector Distance | Time/Frame | #Frames | Frame width | CCDC   |
|--------|---------------|--------|-------|-------------------|------------|---------|-------------|--------|
|        | Bruker        | [K]    |       | [mm]              | [s]        |         |             |        |
| 3a     | D8/ Kryoflex  | Mo     | 100   | 40                | 100        | 735     | 0.55        | 1825002|
| 8      | D8/ Kryoflex  | Mo     | 100   | 35                | 6.4        | 3372    | 0.40        | 1825003|
| 16     | D8/ Oxford    | Mo     | 100   | 50                | 15         | 3532    | 0.35        | 1825004|
| 17b    | D8/ Kryoflex  | Cu     | 100   | 34                | 50         | 1830    | 0.70        | 1825005|
(S,R\*)-2',6'-Diphenyl-3',5,5',7-tetrahydrospro[dibenzo[c,e]azepine-6,4'-dinaphtho[2,1-c:1',2'-e]azepin]-6-ium bromide (3a)

**Figure S1:** Crystal structure of 3a, drawn with 50% displacement ellipsoids. The asymmetric unit is built up by 1 and 2*1/2 independent molecules of 3a. The 2*1/2 molecules, one counter ion and CHCl\(_3\) molecules are omitted for clarity. All three moieties form the same chiral arrangement. The centrosymmetric space group forces the inverse chiral form. Four voids with each 451.2 Å\(^3\) (9.6\% of unit cell) had to be excluded from refinement. The corresponding value of electrons is 109.5 each. We could not find satisfactory positions for solvent atoms.

**Table S3:** Sample and crystal data of 3a.

| Property                        | Value                  |
|---------------------------------|------------------------|
| Chemical formula                | C\(_{50}\)H\(_{38}\)BrCl\(_6\)N |
| Formula weight [g/mol]          | 945.42                 |
| Crystal system                  | monoclinic             |
| Space group                     | C2/c                   |
| Temperature [K]                 | 100                    |
| Z                               | 16                     |
| Measurement method              | \(f\) and \(w\) scans |
| Volume [Å\(^3\)]               | 18676.7(11)            |
| Radiation (Wavelength [Å])      | MoK\(_\alpha\) (\(\lambda = 0.71073\)) |
| Unit cell dimensions [Å] and [°]| 32.9747(11), 90        |
| Crystal size / [mm\(^3\)]      | 0.118 × 0.03 × 0.019   |
| Crystal habit                   | clear colourless needle|
| Density (calculated) / [g/cm\(^3\)] | 1.345                  |
| Absorption coefficient / [mm\(^{-1}\)] | 1.258 |
| Abs. correction Tmin            | 0.6858                 |
| Abs. correction Tmax            | 0.7452                 |
| Abs. correction type            | multiscan              |
| F(000) [e\(^-\)]               | 7712                   |
Table S4: Data collection and structure refinement of 3a.

| Index ranges     | -39 ≤ h ≤ 39, -40 ≤ k ≤ 40, -23 ≤ l ≤ 23 |
|------------------|------------------------------------------|
| Reflections number | 134422                                     |
| Theta range for data collection [°]       | 4.426 to 50.784                            |
| Data / restraints / parameters            | 17153/0/1049                                |
| Refinement method                           | Least squares                              |
| Function minimized                         | Σ w(Fo² - Fc²)                              |
| Final R indices                            |                                          |
| Reflections number                         | 134422                                     |
| Data / restraints / parameters            | 17153/0/1049                                |
| Theta range for data collection [°]       | 4.426 to 50.784                            |
| Reflections number                         | 134422                                     |
| Data / restraints / parameters            | 17153/0/1049                                |
| Reflections number                         | 134422                                     |
| Data / restraints / parameters            | 17153/0/1049                                |
| Reflections number                         | 134422                                     |
| Data / restraints / parameters            | 17153/0/1049                                |
| Reflections number                         | 134422                                     |
| Data / restraints / parameters            | 17153/0/1049                                |
| Reflections number                         | 134422                                     |
| Data / restraints / parameters            | 17153/0/1049                                |
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| Data / restraints / parameters            | 17153/0/1049                                |
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| Data / restraints / parameters            | 17153/0/1049                                |
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| Reflections number                         | 134422                                     |
| Data / restraints / parameters            | 17153/0/1049                                |
| Reflections number                         | 134422                                     |
| Data / restraints / parameters            | 17153/0/1049                                |
2,6-Diiodo-4,5-dihydro-3H-dinaphtho[2,1-c:1',2'-e]azepine (8)

Figure S2: Crystal structure of 8, drawn with 50% displacement ellipsoids. The asymmetric unit is built up by 2 independent molecules of 8. Counter Ion, CHCl₃ and second independent moiety omitted for clarity. The two moieties form different chiral arrangements. Anyhow the chiral space group is proofed by Flack Parameter = 0.000(2).
### Table S5: Sample and crystal data of 8.

| Chemical formula       | C_{24}H_{18}BrCl\_N | Crystal system | monoclinic |
|------------------------|----------------------|----------------|------------|
| Formula weight [g/mol] | 866.8                | Space group    | P21        |
| Temperature [K]        | 100                  | Z              | 4          |
| Measurement method     | \( f \) and \( w \) scans | Volume [\( \text{Å}^3 \)] | 2927.7(3)  |
| Radiation (Wavelength [Å]) | MoKα (\( \lambda = 0.71073 \)) | Unit cell dimensions [Å] and [°] | 11.8178(7) 90 |
| Crystal size / [mm\(^3\)] | 0.253 × 0.217 × 0.204 | 17.2708(10) | 94.2551(19)  |
| Crystal habit          | clear colourless block | Density (calculated) / [g/cm\(^3\)] | 1.967 4.076 |
| Abs. correction Tmin   | 0.6217               | Abs. correction Tmax | 0.746 |
| Abs. correction type   | multiscan            | F(000) [e\(^-\)] | 1648 |

### Table S6: Data collection and structure refinement of 8.

| Index ranges | -16 ≤ h ≤ 16, -24 ≤ k ≤ 24, -20 ≤ l ≤ 20 | Theta range for data collection [°] | 3.456 to 60.186 |
|--------------|------------------------------------------|---------------------------------|-----------------|
| Reflections number | 185276 | Data / restraints / parameters | 17236/1/629 |
| Refinement method       | Least squares | Final R indices | R1 = 0.0229, wR2 = 0.0496 |
| Function minimized      | \( \Sigma w(F_o^2 - F_c^2)^2 \) | L>2σ(1) | R1 = 0.0215, wR2 = 0.0491 |
| Goodness-of-fit on F\(^2\) | 1.048 | Weighting scheme | w=1/[σ\(^2\)(F_o^2)+(0.0231P)^2+2.2240P] |
| Largest diff. peak and hole [e Å\(^{-3}\)] | 1.09/0.68 | where P=(F_o^2+2F_c^2)/3 |
(S,S)-2,6-Diiodo-3,3',5,5'-tetrahydro-4,4'-spirobi[3,1-c:1',2'-e]azepine-ium bromide (16)

Figure S3: Asymmetric unit of 16, drawn with 50% displacement ellipsoids. CHCl₃ omitted for clarity. The chiral space group is proofed by Flack Parameter = 0.059(3).

Table S7: Sample and crystal data of 16.

| Chemical formula | C₄₇H₃₆BrCl₆I₂N | Crystal system | orthorhombic |
|------------------|-----------------|----------------|--------------|
| Formula weight [g/mol] | 1161.18 | Space group | P2₁2₁2₁ |
| Temperature [K] | 100 | Z | 4 |
| Measurement method | \(f\) and \(w\) scans | Volume [Å³] | 4402.1(5) |
| Radiation (Wavelength [Å]) | MoKα (λ = 0.71073) | Unit cell dimensions [Å] and [°] | 8.9851(5) | 90 |
| Crystal size / [mm³] | 0.161 × 0.152 × 0.048 | 11.0959(7) | 90 |
| Crystal habit | clear colourless block | 44.154(3) | 90 |
| Density (calculated) / [g/cm³] | 1.752 | Absorption coefficient / [mm⁻¹] | 2.736 |
| Abs. correction Tmin | 0.6645 | Abs. correction Tmax | 0.747 |
| Abs. correction type | multiscan | F(000) [e⁻] | 2272 |
Table 8: Data collection and structure refinement of 16.

| **Index ranges** | -14 ≤ h ≤ 14, -18 ≤ k ≤ 18, -72 ≤ l ≤ 69 | **Theta range for data collection [°]** | 4.598 to 71.546 |
|------------------|---------------------------------|---------------------------------|-------------------|
| **Reflections number** | 168354 | **Data / restraints / parameters** | 20447/0/523 |
| **Refinement method** | Least squares | **Final R indices** | all data R1 = 0.0360, wR2 = 0.0736 |
| **Function minimized** | Σ w(Fo^2 - Fc^2)^2 | | l>2σ(l) R1 = 0.0318, wR2 = 0.0721 |
| **Goodness-of-fit on F^2** | 1.085 | **Weighting scheme** | w=1/[σ^2(Fo^2)+(0.0296P)^2+5.3285P] |
| **Largest diff. peak and hole [e Å^-3]** | 1.42/-2.32 | | where P=(Fo^2+2Fc^2)/3 |
(R,S*)-2,6-Di(naphthalen-2-yl)-4-(((S,R*)-2'-naphthalen-2-ylmethyl)-[1,1'-binaphthalen]-2-yl)methyl)-4,5-dihydro-3H-dinaphtho[2,1-c:1',2'-e]azepine (17b)

**Figure S4**: Asymmetric unit of 17b, drawn with 50% displacement ellipsoids. CHCl3 omitted for clarity.
Table S9: Sample and crystal data of 17b.

| Chemical formula | C₇₇H₅₇Cl₆N | Crystal system | triclinic |
|------------------|------------|----------------|----------|
| Formula weight [g/mol] | 1208.93 | Space group | P1 |
| Temperature [K] | 100 | Z | 2 |
| Measurement method | f and w scans | Volume [Å³] | 3019.4(5) |
| Radiation (Wavelength [Å]) | CuKα (λ = 1.54178) | Unit cell dimensions [Å] | 12.3238(10) | 71.502(2) |
| Crystal size / [mm³] | 0.259 × 0.198 × 0.098 | | 13.5521(11) | 86.172(4) |
| Crystal habit | clear colourless block | | 19.1746(19) | 84.204(3) |
| Density (calculated) / [g/cm³] | 1.33 | Absorption coefficient / [mm⁻¹] | 2.952 |
| Abs. correction Tmin | 0.6112 | Abs. correction Tmax | 0.7536 |
| Abs. correction type | multiscan | F(000) [e] | 1256 |

Table S10: Data collection and structure refinement of 17b.

| Index ranges | -15 ≤ h ≤ 13, -16 ≤ k ≤ 16, -23 ≤ l ≤ 23 | Theta range for data collection [°] | 6.902 to 146.862 |
|--------------|------------------------------------------|----------------------------------|------------------|
| Reflections number | 31469 | Data / restraints / parameters | 11696/15/766 |
| Refinement method | Least squares | Final R indices | all data | R1 = 0.0750, wR2 = 0.1912 |
| Function minimized | Σ w(Fo² - Fc²)² | | l>2σ(l) | R1 = 0.0707, wR2 = 0.1869 |
| Goodness-of-fit on F² | 1.084 | | | |
| Largest diff. peak and hole [e Å⁻³] | 1.30/-1.38 | Weighting scheme | | w=1/[σ²(Fo²)+(0.0876P)²+5.0094P] |
| | | | where P=(Fo²+2Fc²)/3 |
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