NHC-Promoted Rauhut-Currier Reactions between Vinyl Sulfones and α,β-Unsaturated Aldehydes

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2145 Sheridan Road, Evanston, Illinois 60208

Accessory Publication

General Information ........................................................................................................................................... 2
Procedure for the Synthesis of 1,1-bis(phenylsulfonyl)ethylene 1 ........................................... 2
Spectral data ................................................................................................................................................ 3
X-ray Crystal Structure of 2 ......................................................................................................................... 43
General Information

All reactions were carried out under a nitrogen atmosphere in flame-dried glassware with magnetic stirring. Dichloromethane was purified by passage through a bed of activated alumina. Reagents were purified prior to use unless otherwise stated following the guidelines of Perrin and Armarego. Purification of reaction products was carried out by flash chromatography using EM Reagent silica gel 60 (230-400 mesh). Analytical thin layer chromatography was performed on EM Reagent 0.25 mm silica gel 60-F plates. Visualization was accomplished with UV light. Infrared spectra were recorded on a Perkin Elmer 1600 series FT-IR spectrometer. $^1$H-NMR spectra were recorded on a Bruker A500 (500 MHz) spectrometer and are reported in ppm using solvent as an internal standard (CDCl$_3$ at 7.26 ppm). Data are reported as (ap = apparent, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, b = broad; coupling constant(s) in Hz; integration. Proton-decoupled $^{13}$C-NMR spectra were recorded on a Bruker A500 (125 MHz) spectrometer and are reported in ppm using solvent as an internal standard (CDCl$_3$ at 77.0 ppm). Mass spectral data for the products were acquired on an Agilent 6210 LC-Tof High Resolution Mass Spectrometer, using Electro Spray Ionization source. Samples were introduced using 1uL Direct injection. Solvent used was of 90%MeOH/10% water/ 0.1% Formic acid flowing at 0.5 mL/min.

Procedure for the Synthesis of 1,1-bis(phenylsulfonyl)ethylene 1

Following a procedure from Steinbeck$^3$, to a 100mL round bottom flask containing a magnetic stirring bar was added paraformaldehyde (2.6 g, 28.9 mmol) and methanol (22 mL, 1.3 M). The slurry was then heated to reflux at 80 °C. Once the solution became transparent, the flask was removed from heat and cooled to 0 °C. To the flask was added piperidine (7 mL, 6 mol) at 0 °C and stirred for 15 min. Then, the corresponding arylsulfonyl methane (2.1 g, 7.1 mmol) in 1,4-dioxane (14 mL, 0.5 M) was added dropwise at 0 °C. The reaction stirred for 15 min. Ice water was added to the flask and after 5 min, the solid was collected through vacuum filtration. The white solid (96 %) was dried over P$_2$O$_5$. The white solid was dissolved in benzene in a flame-dried 100 mL round bottom flask. Dry HCl gas was bubbled into the solution until the slurry becomes clear. At this point, the reaction flask was heated to reflux at 80 °C for 3 hrs. The reaction was cooled to 23 °C and the reaction mixture was filtered and concentrated. Recrystallization from benzene/hexanes or ethyl acetate/hexanes produced an off-white solid (91 %). The spectral data matches the literature data.

---

Spectral data

[Structural diagram of a molecule with labels O, H, SO₂Ph, and SO₂Ph]
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Sample Type: Sample  
User Name:  
Acquired Time: 7/14/2011 12:43:16 PM  

ESI Scan (0.140-0.172 min, 3 scans) Frag=230.0V AJ2_07142011_ESI.d Subtract

Exact Mass calcd (M+H)+: 441.0825

441.0825 (M+H)+

458.1087 (M+NH4)+

463.0642 (M+Na)+
3

Cl

SO₂Ph

SO₂Ph

H

O
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**Exact Mass calcd (M+H)+: 475.0435**

+ESI Scan (0.094-0.126 min, 3 scans) Frag=230.0V AJ3_07142011_ESI.d Subtract

| Counts vs. Mass-to-Charge (m/z) | 474 | 475 | 476 | 477 | 478 | 479 | 480 | 481 | 482 | 483 | 484 | 485 | 486 | 487 | 488 | 489 | 490 | 491 | 492 | 493 | 494 | 495 | 496 | 497 | 498 | 499 | 500 | 501 | 502 |
|---------------------------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 475.0438 (M+H)+                 |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |
| 477.0411 (M+H)+                 |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |
| 492.0702 (M+NH4)+               |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |
| 497.0253 (M+Na)+                |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |
| 499.0227 (M+Na)+                |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |

**Diagram:**

- Molecule structure and molecular masses are shown.
- Counts vs. Mass-to-Charge (m/z) range is from 474 to 502.
- Peaks at 475.0438 and 477.0411 are labeled with (M+H)+.
- Peaks at 492.0702 and 497.0253 are labeled with (M+NH4)+.
- Peaks at 499.0227 are labeled with (M+Na)+.
+ESI Scan (0.095-0.112 min, 2 scans) Frag=230.0V AJ4_07142011_ESI.d Subtract

Exact Mass calcld (M+H)+: 491.0981

IRM Calibration Status: Success

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Inj Vol: 0.3
Data Filename: AJ4_07142011_ESI.d

Instrument Name: Instrument 1
Inj Position SampleType: P1-C3 Sample
Position: Sample

User Name: Inj Vol: 0.3

Acquired Time: 7/14/2011 12:48:22 PM

Exact Mass  calcd (M+H)+: 491.0981
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Inj Vol  0.3  InjPosition  AcQ Method  ACQ Method  SampleType  Sample  IRM Calibration Status  Success  
Data Filename  AJ4_07142011_ESI.d  ESI_ASL_Pos_Main_020  Comment  Acquired Time  7/14/2011 12:48:22 PM  

Exact Mass calcd (M+H)+: 491.0981

+ESI Scan (0.096-0.112 min, 2 scans) Frag=230.0V AJ4_07142011_ESI.d Subtract

491.0983  (M+H)+

508.1249  (M+NH4)+

513.0800  (M+Na)+

Counts vs. Mass-to-Charge (m/z)
Br

H

SO₂Ph

SO₂Ph

5

Accessory Publication
Sample Name: aj5  
Position: P1-C4  
Instrument Name: ESI_ASL_Pos_Main_020  
User Name:  

Data Filename: AJ5_07142011_ESI.d  
ACQ Method: ESI_ASL_Pos_Main_020  
SampleType: Sample  
IRM Calibration Status: Success  
Comment:  

Acquired Time: 7/14/2011 12:50:50 PM  

Exact Mass calcd (M+H)+: 518.9930  

Br
O
H
SO2Ph

Exact Mass calcd (M+H)+: 518.9930  

SO2Ph

SO2Ph

207.0989  
350.0888  
730.0792  
922.0953  

Counts vs. Mass-to-Charge (m/z)  

x10^4  

820.0017  

Br
O
H
SO2Ph

Exact Mass calcd (M+H)+: 518.9930  

SO2Ph

SO2Ph

207.0989  
350.0888  
730.0792  
922.0953  

Counts vs. Mass-to-Charge (m/z)
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Instrument Name: Instrument 1

User Name: Inj Vol

Inj Position: Sample

Sample Type: P1-C4

Sample Name: aj5

Sample IRM Calibration Status: Success

Acquired Time: 7/14/2011 12:50:50 PM

**Graph Details:**

- **ESI Scan (0.098-0.114 min, 2 scans) Frag=230.0V AJ5_07142011_ESI.d Subtract**

- Exact Mass calcld (M+H)+: 518.9930

- Peaks at:
  - 518.9942 (M+H)+
  - 530.0517 (M+H)+
  - 536.0262 (M+NH4)+
  - 538.0170 (M+NH4)+
  - 540.9789 (M+Na)+
  - 542.9735 (M+Na)+

Counts vs. Mass-to-Charge (m/z)
Cl

O

H

SO<sub>2</sub>Ph

SO<sub>2</sub>Ph

6
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**ACQ Method**

- **ESI_ASL_Pos_Main_020**

**Comment**

- User Name: Inj Vol: 0.3
- InjPosition: SampleType: Sample
- Acquired Time: 7/14/2011 12:53:23 PM

**Exact Mass**

- **calcd (M+H)+: 475.0435**

**Exact Mass calcld (M+H)+: 475.0435**
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**Data Filename**

AJ6_07142011_ESI.d

**Acquired Time**

7/14/2011 12:53:23 PM

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**Graphical Representation**

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475.0440 (M+H)+

477.0414 (M+H)+

492.0703 (M+NH4)+

497.0268 (M+Na)+

499.0236 (M+Na)+
7

- NC
- SO₂Ph
- SO₂Ph
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**Comment**

User Name: Inj Vol: 0.3

**Inj Position**

Sample Type: Sample

**Sample Name**

aj7

**Position**

P1-C6

**Instrument Name**

ESI_ASL_Pos_Main_020

**IRM Calibration Status**

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**Acquired Time**

7/14/2011 12:55:56 PM

**Acquired Time**

7/14/2011 12:55:56 PM

**Acquired Time**

7/14/2011 12:55:56 PM

**Chart**

+ESI Scan (0.125 min) Frag=230.0V AJ7_07142011_ESI.d Subtract

483.0999

(M+NH4)+

953.1224

Exact Mass calcd (M+H)+: 466.0777

241.1312

610.1768

1418.1863

Counts vs. Mass-to-Charge (m/z)
Sample Name: aj7  Position: P1-06  Instrument Name: Instrument 1  User Name: 

Data Filename: AJ7_07142011_ESI.d  ACQ Method: ESI_ASL_Pos_Main_020  Comment: 

Inj Vol: 0.3  InjPosition:  SampleType: Sample  IRM Calibration Status: Success  Acquired Time: 7/14/2011 12:55:56 PM

**Graph:**

- ESI Scan (0.125 min) Frag=230.0V AJ7_07142011_ESI.d Subtract

Exact Mass calcd (M+H)+: 466.0777

466.0736 (M+H)–

488.0555 (M+Na)+

483.0550 (M+NH4)+

Counts vs. Mass-to-Charge (m/z)
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Acquired Time: 7/14/2011 12:58:28 PM

**Chemical Structure Image:**

- Exact Mass calc'd (M+H)^+: 491.0981
- Exact Mass calc'd (M-H)^-: 491.0981

**Spectrogram Image:**

- ESI Scan (0.102-0.119 min, 2 scans)
- Frag=230.0V
- AJ8_07142011_ESI.d
- Subtract 401.0082 (M+H)^+
- 207.0823
- 349.0889
- 267.1165
- 267.1165
- 700.1866
- 998.2146
O₂N
SO₂Ph
SO₂Ph

\(9\)
O2N
SO2Ph
SO2Ph
O2N
Sample Name: aj9  
Inj Vol: 0.3  
Data Filename: AJ9_07142011_ESI.d  
ACQ Method: ESI_ASL_Pos_Main_020  
User Name: Inj Vol: 0.3  
Sample Type: Sample  
Acquired Time: 7/14/2011 1:01:09 PM  

- ESI Scan (0.087-0.136 min, 4 scans) Frag=230.0V AJ9_07142011_ESI.d Subtract
  + 486.0677 (M+H)+
  503.0547 (M+NH4)+
  508.0463 (M+Na)+

Exact Mass calc'd (M+H)+: 486.0676

![Graphical representation of the mass spectrum]
O
Me
SO₂Ph
SO₂Ph
\[
\text{Me} \quad \text{SO}_2\text{Ph} \\
\text{SO}_2\text{Ph}
\]
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**Inj Vol**: 0.3

**Inj Position**: Sample

**Sample Type**: Sample

**Instrument Name**: ESI_ASL_Pos_Main_020

**Comment**: Success

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*Exact Mass calcd (M+H)+: 483.0931*

![Molecular structure diagram](image)
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**Graph: ESI Scan**

- Exact Mass calcd (M+H)+: 499.0880
- Precursor 516.1146
- Product 1014.1031
- Other peaks: 121.0522, 325.0525, 622.0664, 922.0008, 1120.1464, 1517.2259

Fragmentation: Frag=230.0V AJ11_07142011_ESI.d Subtract
ESI Scan (0.087-0.136 min, 4 scans) Frag=230.0V AJ11_07142011_ESI.d Subtract 151.1146 (M+H+) +

Exact Mass calcd (M+H)+: 499.0880

499.0878 (M+H)+

521.0607 (M+Na)+

Counts vs. Mass-to-Charge (m/z)
X-ray Crystal Structure of 2

A colorless tabular crystal of C23 H20 O5 S2 having approximate dimensions of 0.52 x 0.29 x 0.12 mm was mounted using oil (Infineum V8512) on a glass fiber. All measurements were made on a Bruker APEX-II CCD Diffractometer with a CuKα source at a temperature of 250(2) K with a theta range for data collection of 1.84 to 30.47°. Cell constants and an orientation matrix for data collection corresponded to a Triclinic, space group P-1, with: a = 9.3104(5) Å, b =10.4613(6) Å, c = 11.7985(6) Å, α = 89.825(3)°, β = 70.317(2)° and γ = 72.560(2)°. For Z = 2 and F.W. = 440.51, the calculated density is 1.426 g/cm³. The linear absorption coefficient, μ, for MoKα radiation is 0.293 mm⁻¹. The maximum and minimum transmission factors were: 0.9662 and 0.8625, respectively. Of the 27149 reflections which were collected, 6158 were unique (Rint = 0.0668). The final cycle of full-matrix least-squares refinement on F² was based on 6158 reflections and 271 variable parameters and converged with agreement factors of: R1 = 0.0496 and wR2 = 0.1468 and Goodness-of-fit on F² =0.990. Further information is contained in the CIF file deposited at the CCDC.

The X-ray crystallography data has been submitted to the CCDC. CCDC 833171 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.
checkCIF/PLATON report

You have not supplied any structure factors. As a result the full set of tests cannot be run.

No syntax errors found.  CIF dictionary  Interpreting this report

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Correction method= INTEGRATION

Data completeness= 0.985  Theta(max)= 30.470

R(reflections)= 0.0496( 4533)  wR2(reflections)= 0.1606( 6158)

S = 0.990  Npar= 271

The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

Alert level B

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<th>Alert level</th>
</tr>
</thead>
<tbody>
<tr>
<td>PLAT093_ALERT_1_B</td>
<td>No su's on H-atoms, but refinement reported as .</td>
<td>mixed</td>
<td>B</td>
</tr>
<tr>
<td>PLAT241_ALERT_2_B</td>
<td>Check High Ueq as Compared to Neighbors for C18</td>
<td></td>
<td>B</td>
</tr>
<tr>
<td>PLAT241_ALERT_2_B</td>
<td>Check High Ueq as Compared to Neighbors for C20</td>
<td></td>
<td>B</td>
</tr>
<tr>
<td>PLAT410_ALERT_2_B</td>
<td>Short Intra H...H Contact H4A .. H18 .. 1.81 Ang.</td>
<td></td>
<td>B</td>
</tr>
</tbody>
</table>
Alert level C

ABSTY02_ALERT_1_C  An _exptl_absorpt_correction_type has been given without a literature citation. This should be contained in the _exptl_absorpt_process_details field.

Absorption correction given as integration

PLAT048_ALERT_1_C  MoietyFormula Not Given ........................          ?
PLAT213_ALERT_2_C  Atom C18  has ADP max/min Ratio .....  3.1 prola
PLAT220_ALERT_2_C  Large Non-Solvent C Ueq(max)/Ueq(min) ...  3.8 Ratio
PLAT241_ALERT_2_C  Check High Ueq as Compared to Neighbors for C19
PLAT242_ALERT_2_C  Check Low Ueq as Compared to Neighbors for C5
PLAT242_ALERT_2_C  Check Low Ueq as Compared to Neighbors for C11

Alert level G

PLAT005_ALERT_5_G  No _iucr_refine_instructions_details in CIF ....  ?

0 ALERT level A = Most likely a serious problem - resolve or explain
4 ALERT level B = A potentially serious problem, consider carefully
7 ALERT level C = Check. Ensure it is not caused by an omission or oversight
1 ALERT level G = General information/check it is not something unexpected

3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
8 ALERT type 2 Indicator that the structure model may be wrong or deficient
0 ALERT type 3 Indicator that the structure quality may be low
0 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check

Publication of your CIF

You should attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the nature of your study may justify the reported deviations from journal submission requirements and the more serious of these should be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

If you wish to submit your CIF for publication in Acta Crystallographica Section C or E, you should upload your CIF via the web. If your CIF is to form part of a submission to another IUCr journal, you will be asked, either during electronic submission or by the Co-editor handling your paper, to upload your CIF via our web site.

PLATON version of 27/06/2011; check.def file version of 27/06/2011