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*September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.*

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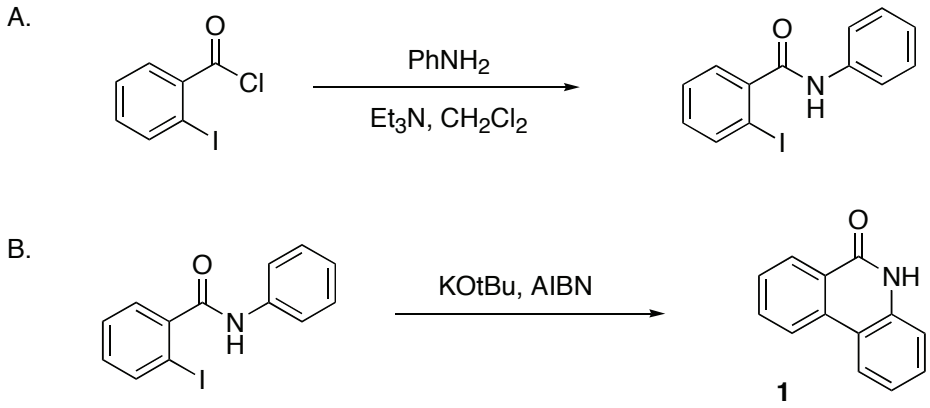
# Potassium *tert*-Butoxide Mediated Synthesis of Phenanthridinone




Submitted by Bhagat Singh Bhakuni, Kaustubh Shrimali, Amit Kumar and Sangit Kumar.[1]*
Checked by David Hughes.

## 1. Procedure

A. *2-Iodo-N-phenylbenzamide*. A 500-mL 3-necked round-bottomed flask, equipped with a 3-cm oval PTFE-coated magnetic stirring bar, is charged with 2-iodobenzoyl chloride (16.3 g, 61.1 mmol, 1.0 equiv) and dichloromethane (80 mL) (Note 1). The center joint is fitted with a 125-mL pressure-equalizing addition funnel equipped with a gas inlet adapter connected to a nitrogen line and a gas bubbler. The other two necks are capped with rubber septa; a thermocouple probe is inserted through one of the septa (Note 2). The flask is immersed in an ice-water bath and the contents cooled to 2 °C. Separately, aniline (7.0 g, 75 mmol, 1.2 equiv) and triethylamine (7.6 g, 75 mmol, 1.2 equiv) are added to a 50-mL Erlenmeyer flask and dissolved in dichloromethane (20 mL). This solution is transferred to the addition funnel then added to the cooled, stirred solution of the acid chloride over 20 min, keeping the internal temperature below 10 °C. The ice-bath is removed and the stirred mixture is allowed to warm to 22 °C and maintained at this temperature for 1.5 h (Note 3). The reaction is worked up by addition of dichloromethane (50 mL) and water (150 mL) and transferred to a 1-L separatory funnel. The layers are separated and the organic layer washed with half-saturated brine, then filtered through a bed of anhydrous sodium sulfate (50 g) in a 150-mL sintered glass funnel into a 500-mL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round-bottomed flask. The sodium sulfate cake is rinsed with dichloromethane (2 x 30 mL). The filtrate is concentrated by rotary evaporation (40 °C water bath, 200 to 10 mmHg) to afford a tan solid (19.1 g). A 4-cm PTFE-coated oval magnetic stir bar and water (150 mL) are added to the flask and the slurry is stirred for 1 h at room temperature. The slurry is filtered through a 150-mL sintered glass funnel, using water (2 x 40 mL) to rinse the flask and wash the filter cake. The product is dried to constant weight in a vacuum oven (50 °C, 70 mmHg, 14 h) to afford 2-iodo-*N*-phenylbenzamide as an off-white crystalline solid (17.6 g, 89% yield) (Notes 4 and 5).

B. *Phenanthridin-6(5H)-one (**1**).* A 1-L 3-necked round-bottomed flask, equipped with a 4-cm oval PTFE-coated magnetic stirring bar, is charged with 2-iodo-*N*-phenylbenzamide (7.0 g, 21.6 mmol, 1.0 equiv), 2,2'-azoisobutyryonitrile (AIBN) (0.77 g, 4.7 mmol, 0.2 equiv), potassium *t*-butoxide (12.0 g, 107 mmol, 5 equiv) and benzene (250 mL) (Notes 6 and 7). The center neck is fitted with a reflux condenser equipped with a gas inlet adapter connected to a nitrogen line and gas bubbler. One outer neck is sealed with a glass stopper; the other neck is capped with a rubber septum through which a thermocouple probe is inserted (Note 2). Using a heating mantle, the slurry is warmed to a gentle reflux (internal temp 81 °C) for 20 h (Notes 8 and 9). An additional portion of AIBN (50 mg, 0.3 mmol, 1.5 %) is added and the mixture refluxed for 5 h (Note 10). The stir bar, reflux condenser and thermocouple probe are removed and the mixture is concentrated by rotary evaporation (40 °C bath temp, 70 to 10 mmHg) to a tan powder (20 g). A 4-cm oval PTFE-coated magnetic stirring bar and water (150 mL) are added to the flask. The suspension is stirred for 1 h at ambient temperature then filtered through a 60-mL sintered glass funnel. Water (2 x 50 mL) is used to rinse the flask and wash the cake. The resulting solid is dried under vacuum (70 mmHg, 40 °C) for 20 h to afford a tan powder (4.04 g) (Note 11). The solid is transferred to a 200-mL round-bottomed flask equipped with a 3-cm oval PTFE-coated magnetic stirring bar. Methyl *t*-butyl ether (MTBE) (40 mL) is added and the slurry is stirred for 1 h at ambient temperature then filtered through a 60-mL sintered glass funnel. The cake is washed with MTBE (2 x 8 mL) then dried under vacuum (70 mmHg, 40 °C) for 4 h to afford phenanthridin-6(5*H*)-one (**1**) as a tan powder (3.59 g, 85% yield) (Notes 12 and 13).

## 2. Notes

1. The following reagents and solvents were used as received for Step A: 2-iodobenzoyl chloride (Alfa Aesar, 98% purity label, but approx. 95% based on $^1$H NMR analysis), aniline (Sigma Aldrich, ACS reagent, 99.5%), triethylamine (Sigma Aldrich, >99.5%), dichloromethane (Fisher, ACS reagent, stabilized).

2. The internal temperature was monitored using a J-Kem Gemini digital thermometer with a Teflon-coated T-Type thermocouple probe (12-inch length, 1/8 inch outer diameter, temperature range –200 to +250 °C).

3. The reaction was monitored by $^1$H NMR. A 0.1 mL aliquot of the reaction mixture was evaporated then dissolved in 1 mL $CDCl_3$. No starting material (< 2%) was detected - diagnostic peaks, starting material δ 8.04-8.10 (m, 2 H); product 7.90 (d, 1 H).

4. 2-Iodo-*N*-phenylbenzamide has the following spectroscopic and physical properties: mp 143–145 °C (lit[2] mp 143–145°C); $^1$H NMR (400 MHz, $CDCl_3$) δ: 7.12–7.20 (m, 2 H), 7.36–7.43 (m, 3 H), 7.61 (br s, 1 H), 7.50 (dd, $J$ = 7.6, 1.2 Hz, 1 H), 7.64 (d, $J$ = 7.9 Hz, 2 H), 7.90 (d, $J$ = 7.9 Hz, 1 H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ: 92.6, 120.3, 125.1, 128.5, 128.7, 129.3, 131.7, 137.7, 140.2, 142.3, 167.4; MS (EI, 70 eV) *m/z* (relative intensity): 323 ($M^+$, 52), 231 ($M^+$- PhNH, 100), 203 (27), 76 (34); IR (plate) $cm^{-1}$: 3310, 1652, 1523, 1441, 1323, 1254, 1013, 739. GC purity: 97% ($t_R$ = 16.5 min; conditions: Agilent DB35MS column; 30 m x 0.25 mm; initial temp 60 °C, ramp at 20 °C/min to 280 °C, hold 15 min).

5. An 87% yield was obtained at half scale.

6. The following reagents and solvents were used as received for Step B: potassium *t*-butoxide (Acros, 98%), AIBN (Sigma Aldrich, 98%), benzene (Fisher, certified ACS, thiophene-free, 99.7%), and methyl *t*-butyl ether (Sigma-Aldrich, ACS reagent, >99%).

7. Five equiv of KO$^t$Bu are essential for complete conversion.

8. Efficient stirring is required since the reaction mixture becomes thick during the early phase of the reaction. If the mixture sets up and stirring stops, the solids should be broken up with a spatula.

9. The reaction was monitored by TLC using 3:7 EtOAc-heptane, $R_f$ = 0.32 for starting material and 0.18 for product. The major by-product, benzanilide, $R_f$ = 0.35 (typical level 4-5%) nearly co-elutes with the starting material. To verify complete conversion, a 0.1 mL aliquot was evaporated then dissolved in DMSO-$d_6$ and analyzed by $^1$H NMR. The N-H protons are

diagnostic (δ 11.7 for phenanthridinone, 10.25 for benzanilide, and 10.4 for 2-iodo-*N*-phenylbenzamide). Other distinguishing peaks include 7.76 (d, 2H) for benzanilide and 7.70 (d, 2H) for 2-iodo-*N*-phenylbenzamide.

10. Additional AIBN is charged if the reaction contains more than 5% unreacted starting material.

11. Based on $^{1}$H NMR analysis, the crude material typically contains 1–2% unreacted starting material, 4–5% benzanilide, and low levels of unknown impurities. The product is nearly insoluble in MTBE and can be readily purified with high recovery by this slurry procedure.

12. Phenanthridin-6(5H)-one (**1**) has the following spectroscopic and physical properties: mp 294–296 °C (lit[3] mp 291–293 °C); $^{1}$H NMR (400 MHz, DMSO-$d_6$) δ: 7.26 (dt, *J* = 1.2, 7.6 Hz, 1 H) 7.37 (dd, *J* = 1.1, 8.1 Hz, 1 H), 7.49 (dt, *J* = 1.1, 7.3 Hz, 1 H), 7.65 (dt, *J* = 1.0, 8.0 Hz, 1 H), 7.87 (dt, *J* = 1.5, 7.2 Hz, 1 H), 8.32 (dd, *J* = 1.1, 8.0 Hz, 1 H), 8.39 (dd, *J* = 1.0, 8.2 Hz, 1 H), 8.51 (d, *J* = 8.2 Hz, 1 H), 11.70 (bs, 1 H). $^{13}$C NMR (100 MHz, DMSO-$d_6$) δ: 116.1, 117.5, 122.2, 122.6, 123.2, 125.7, 127.4, 127.9, 129.5, 132.7, 134.2, 136.6, 160.8; HRMS (ESI) *m/z* 196.0858, calcd for $C_{13}H_9ON + H^+$: 196.0757; MS (EI, 70 eV), *m/z* (relative intensity): 195 ($M^+$, 100), 167 (29), 166 (19), 140 (10), 139 (12), 83 (12); IR (plate), $cm^{-1}$: 3416, 1631, 1360, 1151, 748, 726. GC purity: 99% ($t_R$ = 16.1 min; conditions: Agilent DB35MS column; 30 m x 0.25 mm; initial temp 60 °C, ramp at 20 °C/min to 280 °C, hold 15 min). A wt % purity of 96% was determined by $^{1}$H NMR (with a 10 s pulse delay) using 1,4-dimethoxybenzene as internal standard. Sample preparation: ca. 20 mg of 1,4-dimethoxybenzene and ca 30 mg of product were accurately weighed, then dissolved in 5 mL DMSO-$d_6$. Each of the 8 protons of the product were integrated and compared to the integration of the 6.8 ppm singlet of 1,4-dimethoxybenzene.

13. To avoid the use of benzene, the checker carried out reactions in toluene and in tetrahydropyran (based on a report that THP was an effective solvent for radical reactions, Yasuda, H.; Uenoyama, Y.; Nobuta, O.; Kobayashi, S.; Ryu, I. *Tetrahedron Lett*. **2008**, *49*, 367-370). Neither of these solvents was as effective as benzene, as summarized in the table below. The reactions in toluene were sluggish at 80 °C, with 10–12% unreacted starting material after 50–57 h even with an additional AIBN charge at the 24 h time point, and generated 12 % benzanilide from proto-deiodination. The reactions in THP went to full completion in 20 h, with or without AIBN, but generated 18–23% benzanilide.

| Solvent | Scale (g) | Temp ($^{o}$C) | Time (h) | AIBN (equiv) | SM % | Benzanilide % | Isolated yield[a] |
|---|---|---|---|---|---|---|---|
| Benzene | 7.0 | 81 | 25 | 0.2 | 1.5 | 4 | 85 |
| Benzene | 3.5 | 81 | 25 | 0.2 | 2 | 4 | 83 |
| Toluene | 4.1 | 80 | 57 | 0.3 | 12 | 12 | 62 |
| Toluene | 3.5 | 80 | 50 | 0.3 | 10 | 12 | 68 |
| THP | 3.5 | 85 | 20 | 0.2 | 0 | 18 | 71 |
| THP | 3.5 | 85 | 20 | 0 | 0 | 23 | 61 |

[a]Reactions were purified by MTBE slurry as described in Step B. Products from reactions in THP required two slurry procedures to reduce benzanilide levels to <2%.

## Safety and Waste Disposal Information

The procedures in this article are intended for use only by persons with prior training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011 www.nap.edu). All chemical waste should be disposed of in accordance with local regulations. For general guidelines for the management of chemical waste, see Chapter 8 of Prudent Practices.

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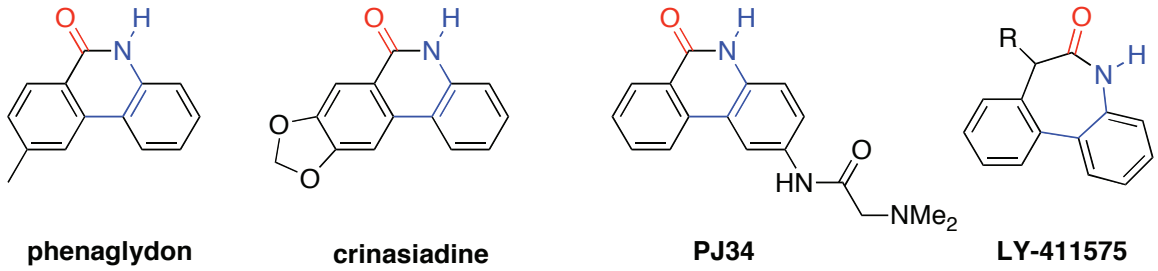
## 3. Discussion

Phenanthridinones and related biaryl lactams are privileged cores present in many alkaloids and pharmaceutically relevant organic molecules (Figure 1).[4] As a result, numerous methods for their syntheses have been described, many involving multiple steps.[5] Palladium-catalyzed biaryl C-C coupling is a key step in most of the reported methodologies, most of which require protection of the N-H bond. A method that avoids a protection/ deprotection strategy and expensive palladium catalyst is highly desirable.

**Figure 1.** Biaryl Lactams




**phenaglydon** **crinasiadine** **PJ34** **LY-411575**

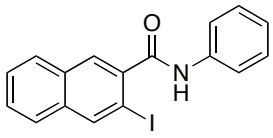
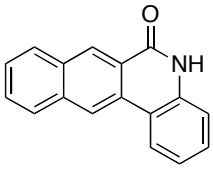
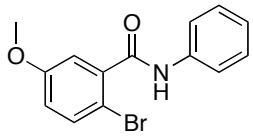
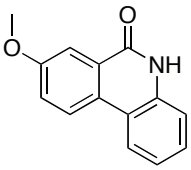
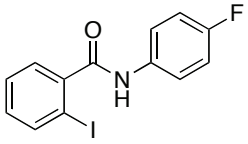
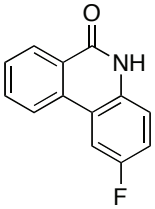
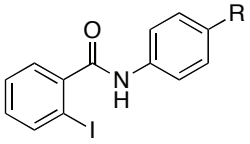
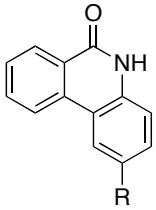
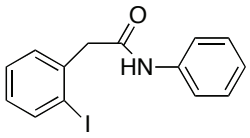
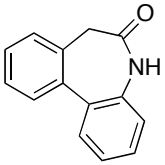
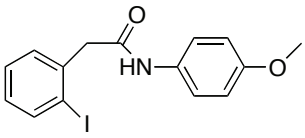
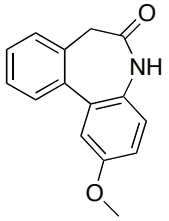
Potassium *t*-butoxide-mediated C-C bond formation for the synthesis of biaryls has been explored by a number of groups.[6] Recently, we reported a carbon-carbon biaryl coupling reaction via C-H activation of anilines for the construction of six-membered phenanthridinones and seven-membered dibenzoazepinone biaryl lactams.[7] Treatment of 2-iodo-*N*-arylbenzamide substrates with potassium *t*-butoxide using catalytic AIBN or 1,10-phenanthroline in benzene solvent gave 81–91% yields of biaryl lactams (Table 1).

KO$^t$Bu alone is not fully effective for the reaction, as only 35% of product was formed in 24 h with 60% unreacted substrate recovered. When 0.2 equiv of the radical initiator AIBN was employed, complete conversion occurred within 24 h and the coupled product was obtained in excellent yield.

Substrates with electron-withdrawing groups, such as fluoride, difluoride, chloride, and bromide, and the electron-donating groups, such as methyl, methoxy, and dimethoxy, on either aryl ring were tolerated under our reaction conditions. Furthermore, the scope of the reaction was extended to other aryl substrates, including naphthalene and thiophene-based 2-halo-arylamide substrates.[7] Regarding the aryl halide, a brief study indicated best yields were obtained with the iodo and bromo substrates (Table 1, entries 1 and 2). The chloride also reacted but gave a moderate yield (49%) of cyclized product.[7]

This metal-free synthesis of phenanthridinones offers several advantages over traditional Pd-catalyzed biaryl couplings with regard to cost, sustainability and lack of toxicity. In addition, the required 2-iodo-*N*-phenylbenzamide substrates are readily accessible by coupling of 2-iodobenzoyl chloride with an arylamine.

**Table 1**. Synthesis of Phenanthridinones and Dibenzoazepinones[a]

| Entry | Amide | Lactam | Additive, Base | Time (h) | Yield (%) |
|---|---|---|---|---|---|
| 1 |  |  | AIBN (0.2 equiv)<br>KO$^t$Bu (5.0 equiv) | 12 | **2** (90) |
| 2 |  |  | AIBN (0.2 equiv)<br>KO$^t$Bu (5.0 equiv) | 12 | **3** (82) |
| 3 |  |  | AIBN (0.2 equiv)<br>KO$^t$Bu (5.0 equiv) | 12 | **4** (81) |
| 4<br>5 |  |  | AIBN (0.2 equiv)<br>KO$^t$Bu (5.0 equiv) | 24<br>24 | **5** R=Me (89)[b]<br>**6** R=OMe (83)[b] |
| 6 |  |  | L (0.2 equiv)<br>KO$^t$Bu (8.0 equiv) | 24 | **7** (91)[c] |
| 7 |  |  | L (0.2 equiv)<br>KO$^t$Bu (8.0 equiv) | 24 | **8** (82) |

[a]Preparation of these compounds is described in reference 7. [b]Two isomers formed in 1:1 ratio, isolated yield for both isomers. [c]Reaction was carried out using 3 g of 2-iodobenzamide substrate following the procedure described herein for **1**. L = 1,10-phenanthroline.

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023, Phone: (+91) 755-409-2325, Fax: (+91) 755-409-2392, E-mail: sangitkumar@iiserb.ac.in

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**3.** Banwell, M. G.; Lupton, D. W.; Ma, X.; Renner, J.; Sydnes, M. O. *Org. Lett*. **2004**, *6*, 2741–2744.

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**6.** (a) Yanagisawa, S.; Ueda, K.; Taniguchi, T.; Itami, K. *Org. Lett*. **2008**, *10*, 4673. (b) Sun, C.-L.; Li, H.; Yu, D.-G.; Yu, M.; Zhou, X.; Lu, X.-Y.; Huang, K.; Zheng, S.-F.; Li, B.-J.; Shi, Z.-J. *Nat. Chem.* **2010**, *2*, 1044. (c) Liu, W.; Cao, H.; Zhang, H.; Zhang, H.; Chung, K. H.; He, C.; Wang, H.; Kwong, F. Y.; Lei, A. *J. Am. Chem. Soc.* **2010**, *132*, 16737. (d) Shirakawa, E.; Itoh, K.-i.; Higashino, T.; Hayashi, T. *J. Am. Chem. Soc.* **2010**, *132*, 15537. (e) Sun, C.-L.; Gu, Y.-F.; Wang, B.; Shi, Z.-J. *Chem. Eur. J.* **2011**, *17*, 10844. (f) Sun, C.-L.; Gu, Y.-F.; Huang, W.-P.; Shi, Z.-J. *Chem. Commun.* **2011**, *47*, 9813.

**7.** Bhakuni, B. S.; Kumar, A.; Balkrishna, S. J.; Sheikh, J. A.; Konar, S.; Kumar, S. *Org. Lett.,* **2012***, 14*, 2838.

## Appendix
## Chemical Abstracts Nomenclature (Registry Number)

2-Iodobenzoyl chloride (2042672)
2-Iodo-*N*-phenylbenzamide (15310-01-7)
Phenanthridin-6(5*H*)-one (2413-02-7)
2,2'-Azoisobutyryonitrile (AIBN) (78-67-1)

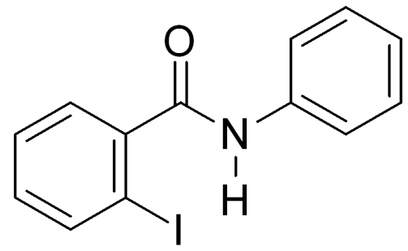
Sangit Kumar was born in Uttar Pradesh, India in 1978, received his PhD degree from Indian Institute of Technology, Bombay in 2004 under the supervision of Professor Harkesh B. Singh. He had first post-doctoral studies with Professor Lars Engman at Uppsala University, Sweden and second post-doctoral experience with Professor Michael R. Detty at University at Buffalo (SUNY), NY, USA. After completing his post-doctoral studies, he returned to India and joined as an assistant professor in the department of Chemistry, Indian Institute of Science, Education, and Research Bhopal. His research interest includes synthesis and biological activity of organochalcogen compounds and transition metal catalyzed carbon-heteroatom coupling reactions

Bhagat Singh Bhakuni was born in Almora, India in 1987. He had completed his graduation in bachelor of science (honors) Chemistry from Kirorimal College of University of Delhi in 2007. Subsequently, he obtained Master Degree in Chemistry from University of Delhi in 2009. He joined the PhD program at Indian Institute Science Education and Research Bhopal in 2009 under the supervision of Dr. Sangit Kumar. Currently he is studying radical chain breaking antioxidant activity of organochalcogen compounds.

Kaustubh Shrimali was born in Maharastra, India in 1990. He cleared 10+2 in year 2007 from S.I.C.A School, Indore, and qualified national level joint entrance exam (IIT JEE) in the year 2008 and was admitted to the BS-MS dual degree program in Indian Institute of Science Education and Research, Bhopal. He is currently pursuing his final year project work under the supervision of Dr. Sangit Kumar. His research interests are organometallics and organic synthesis.

Amit Kumar was born in Uttar Pradesh in 1986, and received his Bachelors of Science in 2006 and Master in Chemistry in 2008 from Maharaj Singh College Saharanpur affiliated with Ch. Charan Singh University Meerut. He joined PhD program in the Department of Chemistry, IISER Bhopal under the supervision of Dr. Sangit Kumar in 2010. He is working on the synthesis of organochalcogen compounds.

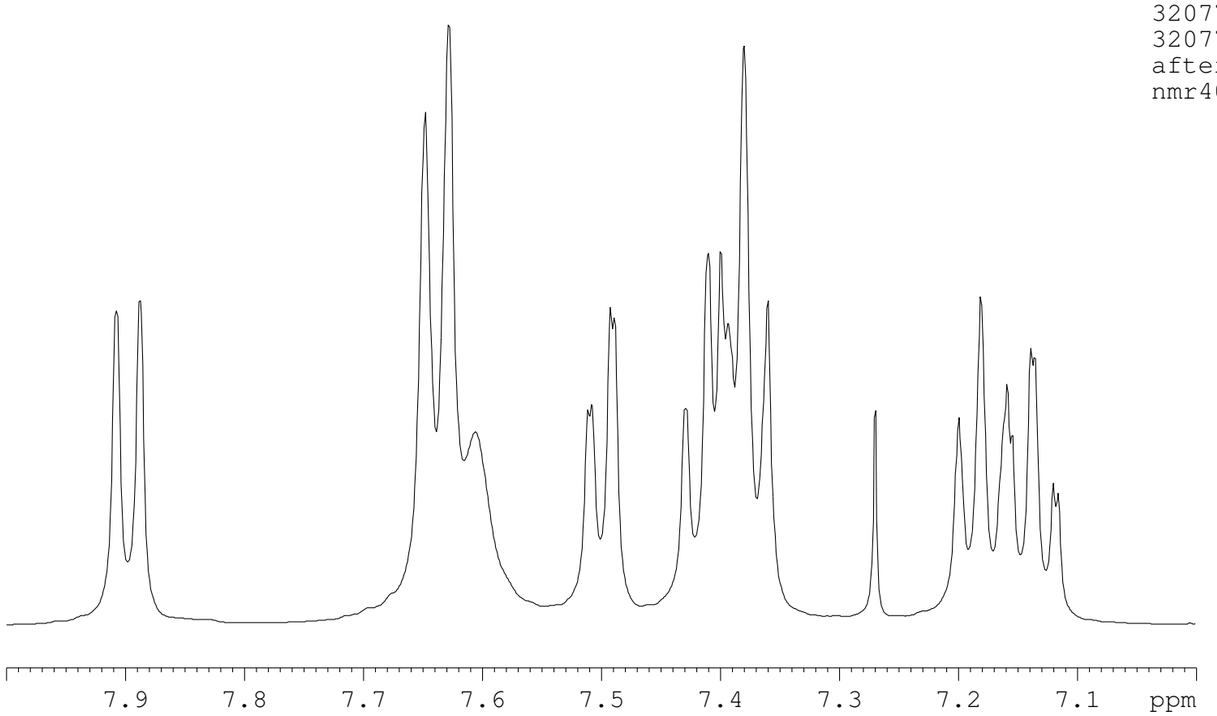
32077-212/2 hughesda
32077-212
after water wash
nmr400b h-1




| | |
|---|---|
| NAME | 32077-212 |
| EXPNO | 2 |
| PROCNO | 1 |
| Date_ | 20121210 |
| Time | 12.49 |
| INSTRUM | spect |
| PROBHD | 5 mm PABBO BB- |
| PULPROG | zg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 32 |
| DS | 2 |
| SWH | 6578.947 Hz |
| FIDRES | 0.200774 Hz |
| AQ | 2.4904180 sec |
| RG | 161.3 |
| DW | 76.000 usec |
| DE | 6.50 usec |
| TE | 300.0 K |
| D1 | 0.10000000 sec |
| TD0 | 1 |

======== CHANNEL f1 ========

| | |
|---|---|
| NUC1 | 1H |
| P1 | 12.75 usec |
| PL1 | -2.00 dB |
| PL1W | 13.05791473 W |
| SFO1 | 400.3324722 MHz |
| SI | 16384 |
| SF | 400.3300035 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |

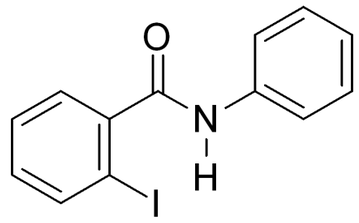






| Peak | ?(F1) [ppm] | ?(F1) [Hz] | Intensity |
|---|---|---|---|
| 1 | 7.9074 | 3165.5695 | 9644605.38 |
| 2 | 7.8877 | 3157.6830 | 9918523.88 |
| 3 | 7.6477 | 3061.6038 | 15242695.94 |
| 4 | 7.6281 | 3053.7573 | 18399040.81 |
| 5 | 7.6057 | 3044.7899 | 5926404.25 |
| 6 | 7.5110 | 3006.8787 | 6614673.81 |
| 7 | 7.5079 | 3005.6376 | 6767832.75 |
| 8 | 7.4921 | 2999.3124 | 9770892.50 |
| 9 | 7.4891 | 2998.1114 | 9431894.56 |
| 10 | 7.4291 | 2974.0916 | 6617786.44 |
| 11 | 7.4102 | 2966.5254 | 11330281.44 |
| 12 | 7.3994 | 2962.2018 | 11453331.50 |
| 13 | 7.3934 | 2959.7998 | 9260793.12 |
| 14 | 7.3802 | 2954.5155 | 17683448.75 |
| 15 | 7.3601 | 2946.4689 | 9701013.44 |
| 16 | 7.2699 | 2910.3591 | 6370430.38 |
| 17 | 7.1994 | 2882.1358 | 5956340.12 |
| 18 | 7.1809 | 2874.7297 | 10074756.44 |
| 19 | 7.1589 | 2865.9225 | 7402702.56 |
| 20 | 7.1546 | 2864.2010 | 5794609.25 |
| 21 | 7.1388 | 2857.8758 | 8495361.12 |
| 22 | 7.1356 | 2856.5948 | 8208934.75 |
| 23 | 7.1199 | 2850.3096 | 4370286.25 |
| 24 | 7.1162 | 2848.8284 | 3813143.69 |

32077-212/3 hughesda
nmr400b c-13




| | |
|---|---|
| NAME | 32077-212 |
| EXPNO | 3 |
| PROCNO | 1 |
| Date_ | 20121210 |
| Time | 13.00 |
| INSTRUM | spect |
| PROBHD | 5 mm PABBO BB- |
| PULPROG | zgdc |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 1032 |
| DS | 4 |
| SWH | 26315.789 Hz |
| FIDRES | 0.401547 Hz |
| AQ | 1.2452340 sec |
| RG | 8192 |
| DW | 19.000 usec |
| DE | 6.50 usec |
| TE | 300.0 K |
| D1 | 0.10000000 sec |
| D11 | 0.03000000 sec |
| TD0 | 40 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 3.50 usec |
| PL1 | 0.00 dB |
| PL1W | 31.90095711 W |
| SFO1 | 100.6741319 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | 120.00 dB |
| PL12 | 17.00 dB |
| PL2W | 0.00000000 W |
| PL12W | 0.16438942 W |
| SFO2 | 400.3320017 MHz |
| SI | 32768 |
| SF | 100.6630405 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| PC | 1.40 |

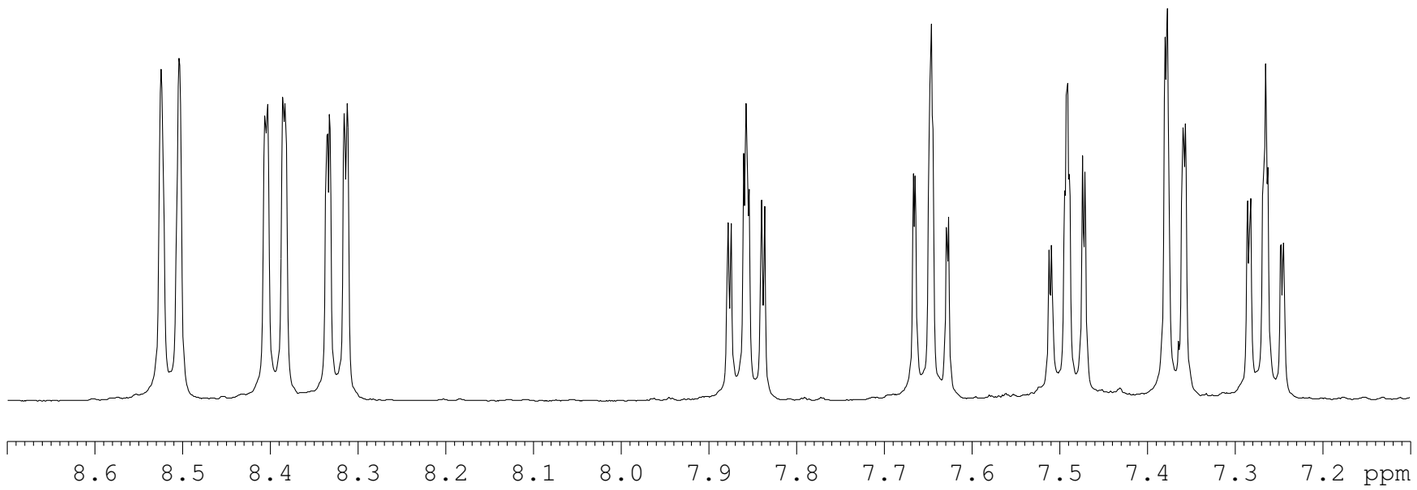
167.43
142.29
140.22
137.74
131.66
129.31
128.72
128.51
125.08
120.33
92.58
77.55
77.44
77.23
76.91

144 142 140 138 136 134 132 130 128 126 124 122 120 118 ppm

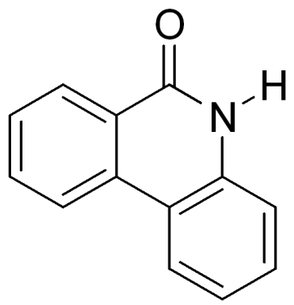
195 190 185 180 175 170 165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 ppm

32077-217/4 hughesda
32077-217
Pure, MTBE slurry
nmr400b h-1

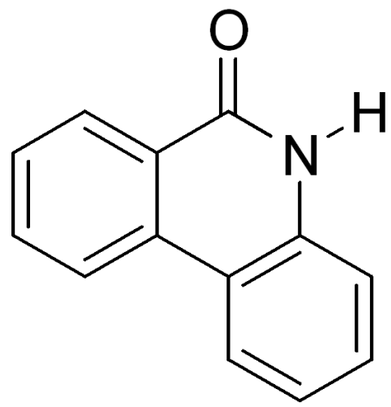
| | |
|---|---|
| NAME | 32077-217 |
| EXPNO | 4 |
| PROCNO | 1 |
| Date_ | 20130118 |
| Time | 11.06 |
| INSTRUM | spect |
| PROBHD | 5 mm PABBO BB- |
| PULPROG | zg30 |
| TD | 32768 |
| SOLVENT | DMSO |
| NS | 64 |
| DS | 2 |
| SWH | 6578.947 Hz |
| FIDRES | 0.200774 Hz |
| AQ | 2.4904180 sec |
| RG | 228.1 |
| DW | 76.000 usec |
| DE | 6.50 usec |
| TE | 293.5 K |
| D1 | 0.10000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 12.75 usec |
| PL1 | -2.00 dB |
| PL1W | 13.05791473 W |
| SFO1 | 400.3324722 MHz |
| SI | 16384 |
| SF | 400.3300000 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |




| Peak | ?(F1) [ppm] | ?(F1) [Hz] | I |
|---|---|---|---|
| 1 | 11.7023 | 4684.7818 | 3294108.8 |
| 2 | 8.5240 | 3412.4129 | 10422777.1 |
| 3 | 8.5037 | 3404.2862 | 10758133.3 |
| 4 | 8.4055 | 3364.9738 | 8958946.00 |
| 5 | 8.4030 | 3363.9730 | 9007430.38 |
| 6 | 8.3852 | 3356.8471 | 9529388.88 |
| 7 | 8.3828 | 3355.8863 | 8780035.56 |
| 8 | 8.3349 | 3336.7105 | 8330325.88 |
| 9 | 8.3321 | 3335.5896 | 9001647.31 |
| 10 | 8.3152 | 3328.8240 | 9020040.0 |
| 11 | 8.3121 | 3327.5830 | 9346144.0 |
| 12 | 7.8780 | 3153.7997 | 5591629.8 |
| 13 | 7.8743 | 3152.3185 | 4364011.9 |
| 14 | 7.8599 | 3146.5538 | 7766177.1 |
| 15 | 7.8569 | 3145.3528 | 9337342.6 |
| 16 | 7.8543 | 3144.3119 | 5826062.5 |
| 17 | 7.8398 | 3138.5071 | 6294013.2 |
| 18 | 7.8360 | 3136.9859 | 4166115.6 |
| 19 | 7.6664 | 3069.0899 | 7131613.5 |
| 20 | 7.6644 | 3068.2893 | 7062897.5 |
| 21 | 7.6465 | 3061.1233 | 9935031.3 |
| 22 | 7.6290 | 3054.1176 | 5453029.0 |
| 23 | 7.6265 | 3053.1167 | 4393968.0 |
| 24 | 7.5119 | 3007.2389 | 4726150.9 |
| 25 | 7.5089 | 3006.0379 | 4880991.4 |
| 26 | 7.4935 | 2999.8729 | 6598792.6 |
| 27 | 7.4913 | 2998.9921 | 9597869.3 |
| 28 | 7.4892 | 2998.1514 | 6953634.8 |
| 29 | 7.4738 | 2991.9864 | 7697486.0 |
| 30 | 7.4708 | 2990.7854 | 7178451.5 |
| 31 | 7.3795 | 2954.2352 | 11424268. |
| 32 | 7.3769 | 2953.1944 | 11557284. |
| 33 | 7.3643 | 2948.1502 | 1871185.3 |
| 34 | 7.3592 | 2946.1085 | 8568997.3 |
| 35 | 7.3565 | 2945.0276 | 8696134.4 |
| 36 | 7.2854 | 2916.5642 | 5342774.2 |
| 37 | 7.2825 | 2915.4032 | 5126040.4 |
| 38 | 7.2651 | 2908.4375 | 10595423. |
| 39 | 7.2626 | 2907.4367 | 6817522.6 |
| 40 | 7.2476 | 2901.4317 | 4863685.3 |
| 41 | 7.2445 | 2900.1907 | 4341219.3 |
| 42 | 2.5112 | 1005.3087 | 6485295.2 |
| 43 | 2.5067 | 1003.5072 | 14145135. |
| 44 | 2.5023 | 1001.7458 | 17759355. |
| 45 | 2.4978 | 999.9443 | 13577777.0 |
| 46 | 2.4935 | 998.2229 | 5939970.38 |



32077-213/6 hughesda
32077-213
slurry in MTBE
nmr400b c-13




| | |
|---|---|
| NAME | 32077-213 |
| EXPNO | 6 |
| PROCNO | 1 |
| Date_ | 20121221 |
| Time | 13.51 |
| INSTRUM | spect |
| PROBHD | 5 mm PABBO BB- |
| PULPROG | zgdc |
| TD | 65536 |
| SOLVENT | DMSO |
| NS | 1817 |
| DS | 4 |
| SWH | 26315.789 Hz |
| FIDRES | 0.401547 Hz |
| AQ | 1.2452340 sec |
| RG | 8192 |
| DW | 19.000 usec |
| DE | 6.50 usec |
| TE | 300.0 K |
| D1 | 0.10000000 sec |
| D11 | 0.03000000 sec |
| TD0 | 40 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 3.50 usec |
| PL1 | 0.00 dB |
| PL1W | 31.90095711 W |
| SFO1 | 100.6741319 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | 120.00 dB |
| PL12 | 17.00 dB |
| PL2W | 0.00000000 W |
| PL12W | 0.16438942 W |
| SFO2 | 400.3320017 MHz |
| SI | 32768 |
| SF | 100.6631091 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| PC | 1.40 |

160.79
136.58
134.22
132.73
129.51
127.86
127.43
125.67
123.19
122.55
122.19
117.52
116.10