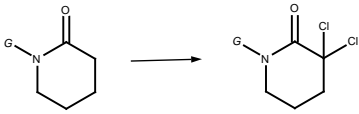
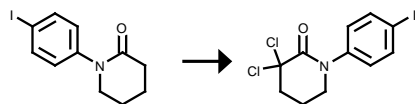
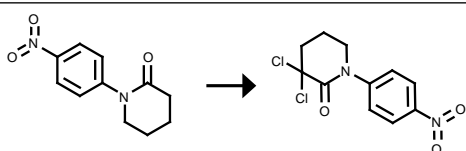


Query

	Query	Results	Date
1. Query	 <p>Search as: As drawn, No salts, No mixtures</p>	8 reactions in Reaxys	2015-04-14 03h:50m:51s (EST)
2. Query	filtered by Reagent/Catalyst	5 reactions in Reaxys	2015-04-14 03h:53m:34s (EST)


 Rx-ID: 10345456 [View in Reaxys](#) 1/5

Yield	Conditions & References
87 %	<p>1.2 :Step 2; 3,3-Dichloro- 1 -(4-iodophenyl)-piperidin-2-one; Phosphorus pentachloride (36.1 g, 174.40 mmol) was added to a solution of 1 -(4-iodophenyl) piperidin-2-one (15.0 g, 49.83 mmol) and chloroform (750 mL). The resulting mixture was heated at reflux for about 3.5 hours, cooled to ambient temperature, and then poured into ice-cold water (200 mL). Standard extractive workup with chloroform (3x400mL), gave the title compound as a pale yellow solid (16.0 g, yield = 87 percent). mp:153-155°C. ¹H NMR (400 MHz, CDCl₃) δ 2.23-2.76 (m, 2H), 2.89-2.92 (m, 2H), 3.73 (t, J= 6 Hz, 2H), 7.04 (d, J= 8.8 Hz, 2H), 7.73 (d, J= 8.4 Hz, 2H) ; IR (KBr) ν 3092, 2949, 2896, 2855, 1679, 1634, 1479, 1310, 1191, 1001, 789, 523 cm⁻¹; MS 370 (M + 1).</p> <p>With phosphorus pentachloride in chloroform, Time= 3.5h, Reflux</p> <p>Patent: AUSPEX PHARMACEUTICALS, INC.; GANT, Thomas, G.; SHAHBAZ, Manoucherhr; WO2010/30983; (2010); (A2) English View in Reaxys</p>
	<p>With phosphorus trichloride in chloroform, Heating</p> <p>Pinto, Donald J.P.; Galembo Jr., Robert A.; Quan, Mimi L.; Orwat, Michael J.; Clark, Charles; Li, Renhua; Wells, Brian; Woerner, Francis; Alexander, Richard S.; Rossi, Karen A.; Smallwood, Angela; Wong, Pancras C.; Luettgen, Joseph M.; Rendina, Alan R.; Knabb, Robert M.; He, Kan; Wexler, Ruth R.; Lam, Patrick Y.S.; Bioorganic and Medicinal Chemistry Letters; vol. 16; nb. 21; (2006); p. 5584 - 5589 View in Reaxys</p>
	<p>With phosphorus pentachloride in chloroform, Heating</p> <p>Pinto, Donald J.P.; Orwat, Michael J.; Quan, Mimi L.; Han, Qi; Galembo Jr., Robert A.; Amparo, Eugene; Wells, Brian; Ellis, Christopher; He, Ming Y.; Alexander, Richard S.; Rossi, Karen A.; Smallwood, Angela; Wong, Pancras C.; Luettgen, Joseph M.; Rendina, Alan R.; Knabb, Robert M.; Mersinger, Lawrence; Kettner, Charles; Bai, Steven; He, Kan; Wexler, Ruth R.; Lam, Patrick Y.S.; Bioorganic and Medicinal Chemistry Letters; vol. 16; nb. 15; (2006); p. 4141 - 4147 View in Reaxys</p>


 Rx-ID: 23754160 [View in Reaxys](#) 2/5

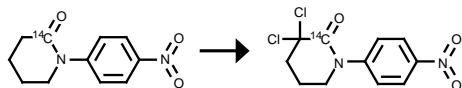
Yield	Conditions & References
	<p>2 :In a dry, water-free 50 gal glass-lined reactor, 27.0 kg PCl₅ was charged followed by 43 kg of chlorobenzene. The slurry was heated to 40° C. with 150 RPM agitation. The above-formed chlorobenzene solution was transferred from the 30 gal reactor to the 100 gal reactor over 7 minutes; the reaction temperature did not exceed 50° C. during the addition. The reactor and transfer line were rinsed forward with 5 kg of chlorobenzene. The reaction mass was heated to 55° C. (15 min) and mixed for one hour. The batch was cooled to 25° C. and sampled for reaction completion. 30 L of purified water was used to rinse the 100 gal glass-lined reactor that was used as a distillate receiver during the vacuum distillation of the lactam reaction mass. Care was taken to ensure water was added to the 100 gal reactor, not the 50 gal, which contained the reaction mixture. Following this rinse, 106 L of purified water was added to the 100 gal reactor, and the water was cooled to 5° C. The reaction mass in the 50 gal reactor was transferred to the cold water quench in the 100 gal reactor so that the quenched mass did not exceed a temperature of 25° C. The transfer time was 40 min. After the transfer was complete, 16 kg n-heptane was charged to the 50 gal reactor to rinse the reactor and the transfer line. This rinse was transferred to the 100 gal reactor, and the contents of the 100 gal reactor were allowed to mix overnight at 66 RPM at ambient temperature. After the overnight hold, 15.4 kg n-heptane was charged to the reactor, and the batch was allowed to mix for 1 hour. A sample was taken and filtered immediately to verify the presence of solid. The batch was filtered on a 36 glass-lined Nutsche filter using a polypropylene bag. The filtration time was 20 minutes. To the 100 gal reactor was charged 21 L of purified water. The water was mixed for 10 minutes to cool and</p>

then was discharged to the Nutsche filter to wash the cake. Next, 16.1 kg n-heptane was charged to the 100 gal reactor, mixed for 10 minutes, and then discharged to the Nutsche filter to wash the cake. The wet cake (10.5 kg) was transferred to drying trays covered with FEP liners. The cake was dried at 50° C. for 28 hours to give 8.5 kg of the desired dichloro product.

With phosphorus pentachloride in chlorobenzene, T= 25 - 55 °C

Patent; Shapiro, Rafael; Rossano, Lucius T.; Mudryk, Boguslaw M.; Cuniere, Nicolas; Oberholzer, Matthew; Zhang, Huiping; Chen, Bang-Chi; US2006/69258; (2006); (A1) English

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Rx-ID: 30495683 [View in Reaxys](#) 3/5

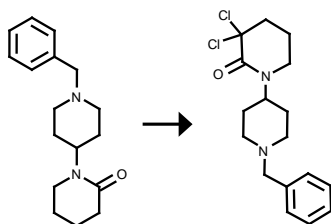
Yield Conditions & References

79 % **With phosphorus pentachloride in chlorobenzene, T= 53 °C**

Maxwell, Brad D.; Tran, Scott B.; Chen, Shiang-Yuan; Zhang, Donglu; Chen, Bang-Chi; Zhang, Huiping; Bonacorsi Jr., Samuel J.; Journal of Labelled Compounds and Radiopharmaceuticals; **vol. 53; nb. 5-6; (2010); p. 363 - 367**
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79 % **With phosphorus pentachloride in chlorobenzene, Time= 1.5h, T= 53 °C**

Maxwell, Brad D.; Tran, Scott B.; Chen, Shiang-Yuan; Zhang, Donglu; Chen, Bang-Chi; Zhang, Huiping; Bonacorsi Jr., Samuel J.; Journal of Labelled Compounds and Radiopharmaceuticals; **vol. 54; nb. 8; (2011); p. 418 - 425**
[View in Reaxys](#)

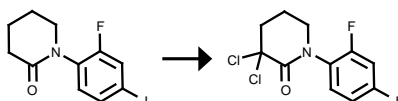


Rx-ID: 10549726 [View in Reaxys](#) 4/5

Yield Conditions & References

85 % **With phosphorus pentachloride in chloroform, Time= 3h, Heating**

Qiao, Jennifer X.; Cheng, Xuhong; Smallheer, Joanne M.; Glemmo, Robert A.; Drummond, Spencer; Pinto, Donald J.P.; Cheney, Daniel L.; He, Kan; Wong, Pancras C.; Luetggen, Joseph M.; Knabb, Robert M.; Wexler, Ruth R.; Lam, Patrick Y.S.; Bioorganic and Medicinal Chemistry Letters; **vol. 17; nb. 5; (2007); p. 1432 - 1437**
[View in Reaxys](#)



Rx-ID: 10462743 [View in Reaxys](#) 5/5

Yield Conditions & References

With phosphorus pentachloride in chloroform, Heating

Pinto, Donald J.P.; Orwat, Michael J.; Quan, Mimi L.; Han, Qi; Glemmo Jr., Robert A.; Amparo, Eugene; Wells, Brian; Ellis, Christopher; He, Ming Y.; Alexander, Richard S.; Rossi, Karen A.; Smallwood, Angela; Wong, Pancras C.; Luetggen, Joseph M.; Rendina, Alan R.; Knabb, Robert M.; Mersinger, Lawrence; Kettner, Charles; Bai, Steven; He, Kan; Wexler, Ruth R.; Lam, Patrick Y.S.; Bioorganic and Medicinal Chemistry Letters; **vol. 16; nb. 15; (2006); p. 4141 - 4147**

