HANDS ON CHEMISTRY REVIEW

Issue 3, 2011

Welcome to Hands on Chemistry Review

"Hands on Chemistry Review" is the newsletter of Digital Specialty Chemicals. Digital Specialty Chemicals Limited is a manufacturer of high quality organophosphorus and organometallic chemicals that are in demand in the global pharmaceutical, specialty chemical and semiconductor markets. DSC offers materials in kilogram to metric ton quantities. In each issue we bring you company news, employee biographies, new and developmental product information, technical reports and notice of upcoming events. Any questions or suggestions you have regarding the newsletter should be directed to Bill Stibbs on +1-(416)231-2991x13 or marketing@digitalchem.ca. To subscribe to the electronic version please send your e-mail address to marketing@digitalchem.ca. Please also contact Bill to obtain access to previous issues of Hands on Chemistry Review. We would be happy to interact with you on a more frequent basis: please see the links to our social media sites at the end of the newsletter.

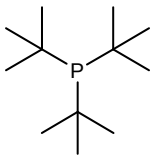
Upcoming Tradeshow

We will be exhibiting at CPhI Worldwide, 25-27th October in Frankfurt (stand F0H11, Forum 0, Fine Chemicals Zone). For the first time we will have our own stand in the Fine Chemicals sector and would like to invite our clients to stop by to review our latest offerings.

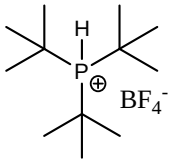
Product Review

Scott A. Laneman, Ph.D., Scientific Fellow

Tri(tert-Butyl)phosphine and its' HBF_4 Salt



Product Number: 839
CAS#: [13716-12-6]
$C_{12}H_{27}P$
Molecular Weight: 202.32



Product Number: 1691
CAS#: [131274-22-1]
$C_{12}H_{28}BF_4P$
Molecular Weight: 290.13

Tri(tert-butyl)phosphine, (t-Bu)$_3$P, and tris(tert-butyl)phosphonium tetrafluoroborate, (t-Bu)$_3$P•HBF_4, are used as bulky electron rich phosphines in many palladium-catalyzed reactions. (t-Bu)$_3$P is a very air-sensitive (pyrophoric) low melting solid (mp=30°C), which requires strict air-exclusion. (t-Bu)$_3$P storage is recommended at low temperatures under inert atmosphere (nitrogen or argon). (t-Bu)$_3$P is often stored in a solution (toluene, hexane, etc.), which allows for easier handling and manipulation. (t-Bu)$_3$P is a very basic phosphine with a pK_a of 11.4.[1] (t-Bu)$_3$P can be stored and used as the air and moisture stable HBF_4-phosphonium salt. The lone pair of (t-Bu)$_3$P•HBF_4 can be "deprotected" *in situ* with addition of base. (t-Bu)$_3$P•HBF_4 is stable upon exposure to air at room temperature for >4 months.[1] Pd/(t-Bu)$_3$P catalyst systems were groundbreaking in that inexpensive aryl chlorides could be used as coupling partners instead of aryl bromides and iodides.[2-4]

This technical summary will highlight only a few of the many catalytic transformations that utilize (t-Bu)$_3$P and (t-Bu)$_3$P•HBF_4.

The phosphine ligand and its' HBF_4 salt are both available from DSC. Should you prefer the ready to use palladium catalyst this is available from our partner, Umicore.

Suzuki Coupling of Aryl Halides

Pd/P(tBu)$_3$ catalysts are capable of coupling a wide variety of aryl coupling partners (chlorides, bromides, iodides, and sulfonyl chlorides).[5] Interestingly, Pd/P(tBu)$_3$ catalysts are not reactive towards triflate groups unlike Pd/PCy_3 catalysts.[2] Inexpensive aryl chlorides are of interest. Both activated chlorides (aryl chlorides with electron withdrawing groups) and unactivated chlorides (aryl chlorides with electron donating groups) are suitable coupling partners. The most active catalysts contain a ratio of Pd/P(tBu)$_3$ of 1:1. In unactivated aryl chlorides, the combination of electron-rich and steric qualities of P(tBu)$_3$ give Pd complexes activity. The electron-richness of these Pd/P(tBu)$_3$ complexes allow oxidation-addition of the Ar-Cl bond. The steric demand facilitates dissociation to the reactive mono-phosphine/Pd adduct.[2]

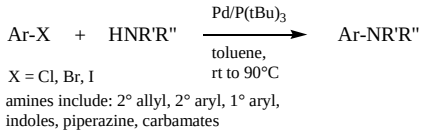
$$\text{Ar-X} + \text{Ar'-B(OH)}_2 \xrightarrow{\text{Pd/P(tBu)}_3} \text{Ar-Ar'}$$

X = Cl, Br, I, SO_2Cl

Buchwald-Hartwig Amination of Aryl Halides

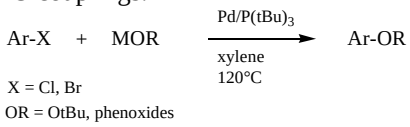
Pd/P(tBu)$_3$ catalysts couple unactivated chlorides with secondary amines at 70°C while activated aryl chlorides

and chlorobenzene react at room temperature. Improved rates were observed with a catalyst ratio of 1/0.8 (Pd/P(*t*Bu)$_3$). Aryl chlorides with ortho-substituents are not suitable substrates in C-N coupling.[6-8]



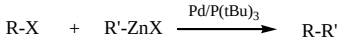
Carbon-Oxygen Coupling of Aryl Halides

NaO*t*Bu and Na-phenoxides are useful coupling partners, but aryl chlorides that lack ortho-substitution are not suitable partners. The ortho-substitution may be required for steric acceleration of the turnover-limiting reductive elimination step. Electron neutral aryl chlorides can couple with NaO*t*Bu. Alcohols that contain β-hydrogen atoms cannot be used in C-O couplings.[9,10]

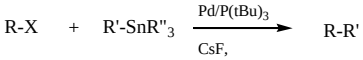


Carbon-Carbon Bond Coupling of Aryl and Vinyl Substrates via Negishi (Zn), Stille (Sn), and Kumada (Mg) Reactions

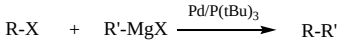
Pd/P(*t*Bu)$_3$ catalysts can couple aryl and vinyl chlorides and sulfonyl chlorides with zinc reagents to form C-C bonds (Negishi reaction). This catalyst system is suitable for unactivated and electron-rich chlorides. Similar to other Pd/P(*t*Bu)$_3$ systems, a ratio of 1:1 is much better than 1:2 (Pd/P(*t*Bu)$_3$).[11] Arylation of zinc enolates have been reported.[12] Stille coupling of organotin reagents with aryl chlorides and bromides have been reported with Pd/P(*i*Bu)$_3$/CsF catalyst systems. These couplings proceed with electron deficient, electron-rich, and hindered aryl chlorides. Catalysis with aryl chlorides proceeds at 100°C while aryl bromides react at room temperature. Aryl chlorides selectively react under these catalytic conditions in the presence of triflates.[13-15] Limited Kumada couplings of aryl bromides and iodides with Grignard reagents have been reported; however, the numbers of examples were limited due to the incompatible of Grignard reagents with functionalized substrates.[16]



X = Cl, Br, I, SO_2Cl
R = aryl, alkenyl
R' = aryl, acetamides



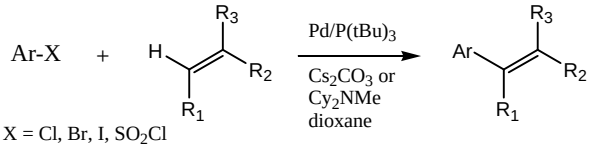
X = Cl, Br
R = aryl
R' = aryl, alkenyl, alkyl, allyl
CsF, dioxane, rt to 100°C



X = Br, I
R and R' = aryl

Mizoroki-Heck Carbon-Carbon Bond Coupling of Aryl and Vinyl Substrates

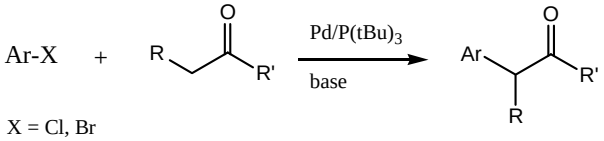
A wide variety of Pd catalysts have been successfully used with/without ligands for this type of coupling. Pd/P(*t*Bu)$_3$ catalysts with Cs_2CO_3 are effective in the coupling of electron-rich and hindered aryl chlorides with styrene or methyl acrylates. Replacement of Cs_2CO_3 with Cy_2NMe allowed reactions to proceed at room temperature instead of >100°C. Also, the use of Cy_2NMe increased the scope of the olefin partner (monosubstituted and disubstituted olefins). Unactivated aryl chlorides can be coupled, as well as heteroaryl chlorides.[17-20]



X = Cl, Br, I, SO_2Cl
Ar = aryl, heteroaryl

α-Arylation of Aldehydes, Ketones, and Esters with Aryl Halides

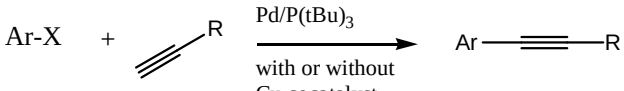
Pd/P(*t*Bu)$_3$ catalysts can successfully α-arylate aldehydes, ketones, and esters, which include malonates and protected amino esters. Electron-rich and hindered aryl chlorides can be used as coupling partners while certain electron-withdrawing groups (esters, ketones, nitriles) are not well tolerated.[7,21-23]



X = Cl, Br
Ar = aryl, heteroaryl
R = aryl, $-CO_2R"$, alkyl, $-N=CPh_2$, CN
R' = H, alkyl, -OR"

Sonogashira Reaction of Aryl Halides and Sulfonyl Chlorides with Terminal Alkynes

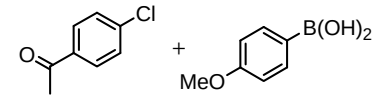
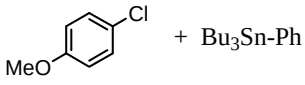
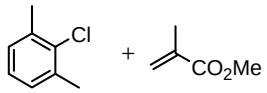
Terminal alkynes cleanly couple with aryl bromides and chlorides in the presence of Pd/P(*t*Bu)$_3$ and Cu catalysts. Electron-rich aryl bromides couple at room temperature. The Sonogashira coupling of aryl bromides can be achieved without Cu cocatalysts. Aryl chlorides and aryl sulfonyl chlorides couple at elevated temperate and in the presence of Cu cocatalysts.[24-26]



X = Cl, Br, SO_2Cl
Ar = aryl, heteroaryl
R = aryl, alkyl, alkenyl, silyl

Use of P(tBu)$_3$•HBF$_4$ in Catalytic Reactions

(t-Bu)$_3$P•HBF$_4$ is an air stable form of (t-Bu)$_3$P and allows for easy handling. Several reports have demonstrated the use of (t-Bu)$_3$P•HBF$_4$ in catalytic processes: *in situ* Pd complexes being formed upon addition of a base. A few direct comparisons of Suzuki, Stille, and Mizoroki-Heck couplings with (t-Bu)$_3$P and (t-Bu)$_3$P•HBF$_4$ are shown below.[4] The selection of base was not important. Most Pd/P(tBu)$_3$ catalyzed reactions required base, which was added in slight excess to deprotonate (t-Bu)$_3$P•HBF$_4$ back to (t-Bu)$_3$P.

	P(tBu)$_3$	P(tBu)$_3$•HBF$_4$
	93%	90%
	94%	93%
	80%	90%

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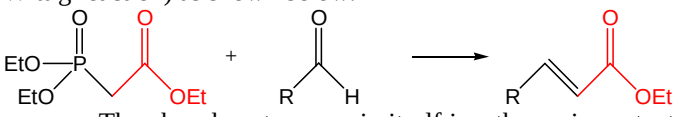
Methyl or Ethyl? Does it Make a Difference?

The choice of these two simple starting materials may have a huge impact in the economics of your potential blockbuster process. Digital would like to make our customers aware with this article of the importance in the choice between Dimethyl Phosphonate and Diethylphosphonate. This simple choice at the R&D stage can eventually lead to significant supply and price issues during larger scale up.

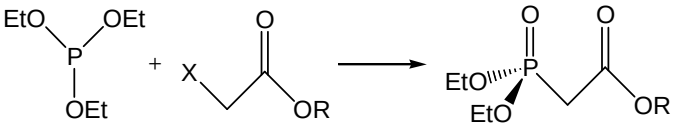
Many companies spend significant amounts of time and effort in the research of new molecules to develop new pharmaceutically active substances or fine chemicals with very specific properties. DSC itself spends considerable amounts of money every year to help our customers with new substances. As such, Digital is aware that our customer's R&D Departments require speedy service and are under considerable pressure to develop new products. We are also aware that customers may have several different synthetic approaches. As a new product reaches the commercial stage, development timeframes become crunched. At this stage, reliable supply and cost structures suddenly play a major role in the selection of a suitable supplier. DSC has observed in many cases that the quick initial decision at our customer's R&D departments might solve the initial project problems, but, these decisions can result in significant costs and operational issues during scale up to pilot and production levels.

The example below illustrates the issues that involve a simple ethyl or methyl group: our pharma customers find phosphonate esters made by DSC generally useful to convert a carbonyl group (C=O) into an olefin (C=C, analogue to the Wittig reaction) as shown below.



The phosphonate group in itself is rather unimportant. The key part of the molecule is the "C-rest" (in red) connected to the P- Atom. Eventually, this will provide the functionality to our customers' downstream chemistry. The phosphorus group turns into a waste by-product. As such it doesn't matter in the end whether a customer uses a dimethyl or a diethyl phosphonate (there may be an argument about volume/price efficiency that slightly prefers the Methyl over the Ethyl group).

An average chemist with limited scale-up experience might just prefer the dimethyl phosphonate since they are readily available at catalogue companies. This chemist usually does not recognize that there is a significant difference in the manufacture of the Phosphonate ester. Phosphonate Esters are manufactured by the Arbuzov reaction:



This reaction uses phosphites and organic chlorides, bromides or iodides, with preference given to chlorides because of cost/price reasons. This is in particular important at larger volumes. The reaction leading to the diethyl phosphonates will generate ethyl chloride or bromide as shown above, which usually is no problem as ethyl chloride or bromide are liquids with low boiling points. The same reaction starting with trimethylphosphite to make the dimethyl phosphonate generates methyl bromide or methyl chloride. Methyl chloride is a gas at room temperature with a boiling point of -24°C and is difficult to trap as an off-gas. Compounding the problem, methyl chloride is a chlorohydrocarbon and categorically cannot be vented into the atmosphere. Also, very few companies have incinerators that can deal with this kind of off-gas at large scale.

At low volumes for lab trials where price is usually no problem, DSC would explore the organic iodide to generate methyl iodide (a liquid at room temperature) in the event our customers request specifically the dimethyl phosphonate. However, if price and availability becomes an issue at larger volumes, Digital feels obligated to inform our customers early on that they will find it much more difficult to find suppliers of dimethly phosphonates than the diethyl phosphonates based on the reasons above.

Hopefully, you will find this article useful in future in making the right choice between Methyl and Ethyl!

Facility Expansion

DSC has built a reputation of being a respectable supplier of low volume Specialty Chemicals for developmental projects at our customer base through our technical expertise (approximately one third of us have PhD's) and the entrepreneurial spirit of the majority owner Ravi Gukathasan. This involvement in developmental products gives DSC access to a significant product pipeline. In order to support our traditional customers with the volumes required as their projects grow we have recently completed a significant expansion of our manufacturing facility.

The centerpiece of the expansion is a 1,200L glass-lined steel reactor with glass overheads and a heater / chiller supporting operating temperatures of -40 to +200°C under inert atmosphere, shown above. This reactor increases our manufacturing volume by 30%. The planning for a second 1,200L reactor including auxiliary equipment is well under way. We have also expanded our fractional distillation capacity with a 120L stainless steel unit to support increased volumes in our high purity organometallics business. In 2012 we will be commissioning a hybrid molecular distillation unit with a rectifying column to facilitate the manufacture of ultra-high purity, heat-sensitive liquids.

In addition, DSC continues to make a significant investment in the development of new products: the life blood of our organization. We are in the process of a significant expansion to our R&D department with the addition of two chemists, several benchtop and walk-in fumehoods, and five 10L reactors.

Challenge the Team!

DSC has a good market reputation for developing, scaling up and supplying new products where phosphorus plays a dominant role. A number of examples can be found in this newsletter.

We also venture into new areas where we can apply the knowledge of organometallic chemistry developed in-house to successfully work on a wider variety of compounds. One such example is the electronics market which is in need of volatile high purity organometallic products for semiconductor applications. DSC has gained some fame over the last few years in this market area - in some cases attaining a market leader position.

As a relatively small but agile organization with no corporate bureaucracy, DSC is constantly scouting the market for new products that are in demand, even outside our current product portfolio. We're always interested to hear from our customers if they have difficulties sourcing particular raw materials or would like to establish a second or third supplier. These products become even more interesting for DSC if they fall into our preferred volume range of 10-1000Kg, if the supply is scarce and if there is a continuous demand.

DSC welcomes all inquiries and we usually reply within three working days as to whether the product in question is viable for DSC and often have a price quote available, provided there are not too many production steps involved and raw materials are freely available.

Challenge the DSC team by forwarding your inquiries to marketing@digitalchem.ca.

Marketing Partnership with evocatal

DSC recently entered a marketing agreement with evocatal. Below is an overview of evocatal's products and technologies.

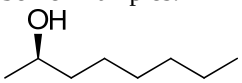
The Germany-based company focusses on the development and production of chiral biocatalysts. Biocatalysis is today the most efficient way to establish robust and economic processes for large scale manufacturing of chiral fine chemicals. Mastering unique molecular technologies and experienced in commercial production processes, evocatal has developed a number of highly efficient biocatalysts including:

- Ketoreductases (ADHs/KREDs) for the synthesis of chiral alcohols
- Aminotransferases (Transaminases) for the synthesis of chiral amines
- Lipases for chiral resolution and esterifications
- Hydroxynitrile lyases as chiral cyanhydrin-intermediates
- Cofactor regeneration systems minimizing the cost of synthesis

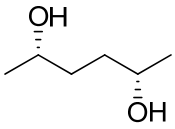
Based on this growing product portfolio, evocatal has entered the fine chemicals market in 2010. We are offering a range of chiral secondary alcohols, aliphatic diols, hydroxyesters/-ketones, phenylethanols in quantities up to multi-ton scale. Based on our fruitful collaborations in the past, Digital Specialty Chemicals will be the supplier of evocatal's chemicals in North America.

Compounds are applied, for example, as building blocks in API manufacturing processes for the Pharmaceutical and Agrochemical industries. Chiral diols are used as ligands to manufacture chemical catalysts like DuPhos, which will be off patent in 2012 worldwide. Also, the electronics industry is a vast market for chiral products, for example, to synthesize liquid crystals.

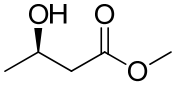
Some Examples:



(*R*)-(-)-2-Octanol: used as a building block for liquid crystals



(*S,S*)-2,5-Hexanediol: applied as a ligand for chemical catalysts



Methyl (*R*)-3-Hydroxybutyrate: known building block for Dorzolamide synthesis

Based in Duesseldorf, evocatal not only grows its catalog business but also tailors catalysts and develops production processes as a CMO/CRO service. The business flourishes, as the technology meets the demands of the modern chemical industry in terms of sustainability and environmentally clean production processes.

We look forward to keeping you updated about our latest products and new technologies. Please visit our website www.evocatal.com or contact DSC (sales@digitalchem.com) for more information.

Contract Service Offerings

DSC offers custom manufacturing and toll distillation services based on our expertise and technology in the areas of high throughput distillation of heat-sensitive liquids and high pressure hydrogenation supported by our molecular distillation equipment and Parr pressure reactors. Inquiries about these services should be directed to Bill Stibbs, Director of Marketing, marketing@digitalchem.ca or your sales manager.

Employee Biography: Christine Harnett, Manager, US Sales & Business Development

Christine recently joined the DSC global sales team and is managing sales in the U.S. to the pharmaceutical industry. After obtaining a B.S. in Biochemistry from the University of Massachusetts and after some post-graduate courses at Purdue University, Christine worked in biochemical research at the present Dana Farber Cancer Institute in Boston, MA. In this role, she interacted with equipment sales representatives and thought that combining science and business would be a fun and rewarding career path. She completed a MBA program at Northeastern University with an emphasis in marketing.

Her first sales position was to market gel electrophoresis and related equipment to protein and nucleic acid scientists at medical research centers, in academia and in industry. She transitioned to working out of a virtual office and traveling to the customers' sites for meetings and demonstrations. Christine quickly realized that she liked the flexibility, independence and enhanced productivity which the home office arrangement offered. She broadened her experience by joining a small biotech company which developed and produced living skin equivalents as replacements for burn patients and for product safety testing. This position offered Christine the opportunity to do technical service and sales in Europe and the U.S. She says, "I enjoyed the interactions with all the international customers and learned some traditions uncommon in America such as the knuckle knocking applause on the table in Germany and adding melted cheese to potato fries in the Netherlands."

After working for the biotech company, Christine started her business development career in chemical sales and contract manufacturing. She worked at the catalog companies, Sigma Aldrich and Lancaster Synthesis where she sought new clients for bulk and custom chemicals in the pharmaceutical, medical diagnostic, industrial and electronic markets. After Lancaster Synthesis, Christine worked for The Dow Chemical Company in the Dowpharma global business unit. At Dowpharma, she was responsible for marketing cGMP contract manufacturing services to pharmaceutical companies of all sizes. This position also offered the opportunity to sell products such as activated mPEGs (methoxy PolyEthylene Glycols) to large molecules customers and chiral conversion solutions to small molecule customers. Dowpharma, UK was purchased by Dr. Reddy's Laboratories in 2008 and was incorporated into the custom pharmaceutical services group. Christine's responsibilities at Dr. Reddy's Laboratories expanded to include offering generic APIs and formulation services to innovator pharmaceutical companies.

Christine is excited to be a part of the DSC team and looks forward to contributing to the company's financial growth by helping existing customers with additional products and by finding new clients for the company's products and custom manufacturing services. In her leisure time, Christine enjoys playing golf and bicycle riding and strives to keep her EMT certificate current.

Community Involvement

We recently held our annual company picnic. As you can see a great time was had by our employees and their families.

We also held a barbecue to celebrate Canada Day (1st July) and were a proud sponsor of the Family Fun Day organized by the West Hill Social Activity Club on 20th August.

Bill Stibbs, Director of Marketing, presented the Digital Specialty Chemicals Graduate Scholarship in Chemistry to Paraskevi Lagaditis at the University of Toronto, Chemistry Department's Awards Reception. Paraskevi's research is focussed on chiral iron complexes and their potential as catalysts for asymmetric transfer hydrogenation (Photograph courtesy of University of Toronto, Department of Chemistry).

DSC, and our employees, has been raising funds for the drought in the Horn of Africa. Donations are being made to "World Vision Canada".

Pot Luck Lunch Recipe

The pot luck lunch (this is a lunch where all the participants bring one dish and everybody shares the food) is a regular part of our social program. Below is a recipe from Caroline Schweitzer, our Customer Service Manager, who often takes the prize for best dessert. We hope you will not only try it, but enjoy it as much as we did!

Raspberry and White Chocolate Mousse Trifle

Raspberry Sauce

2 pints fresh or 1 600g bag frozen raspberries, thawed
2 tbsp sugar, juice of 1 lemon

White Chocolate Mousse

12 oz white chocolate, chopped
½ tsp vanilla extract
2 tsp brandy
1 fresh egg, slightly beaten and room temperature
1 ¼ cup heavy cream, 1 cup is whipped
1 ½ lb pound cake, cut into ½" pieces
Semi-sweet chocolate or fresh raspberries to decorate

Assembly

Sauce: Puree the raspberries with sugar and lemon juice. Strain through a sieve to remove the seeds. Refrigerate until ready to use.

Mousse: Over simmering water, warm 1/4 cup of the cream and the white chocolate, stirring constantly until chocolate melts. Let mixture cool until it is lukewarm and add the egg, vanilla and brandy. Fold whipped cream into melted chocolate mixture, one-third at a time, until no streaks remain. Refrigerate at least 1 1/2 hours.

Assembly: Layer cake, raspberry puree and mousse in a bowl three times. Refrigerate several hours or overnight. Decorate with fresh raspberries or chocolate curls.

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