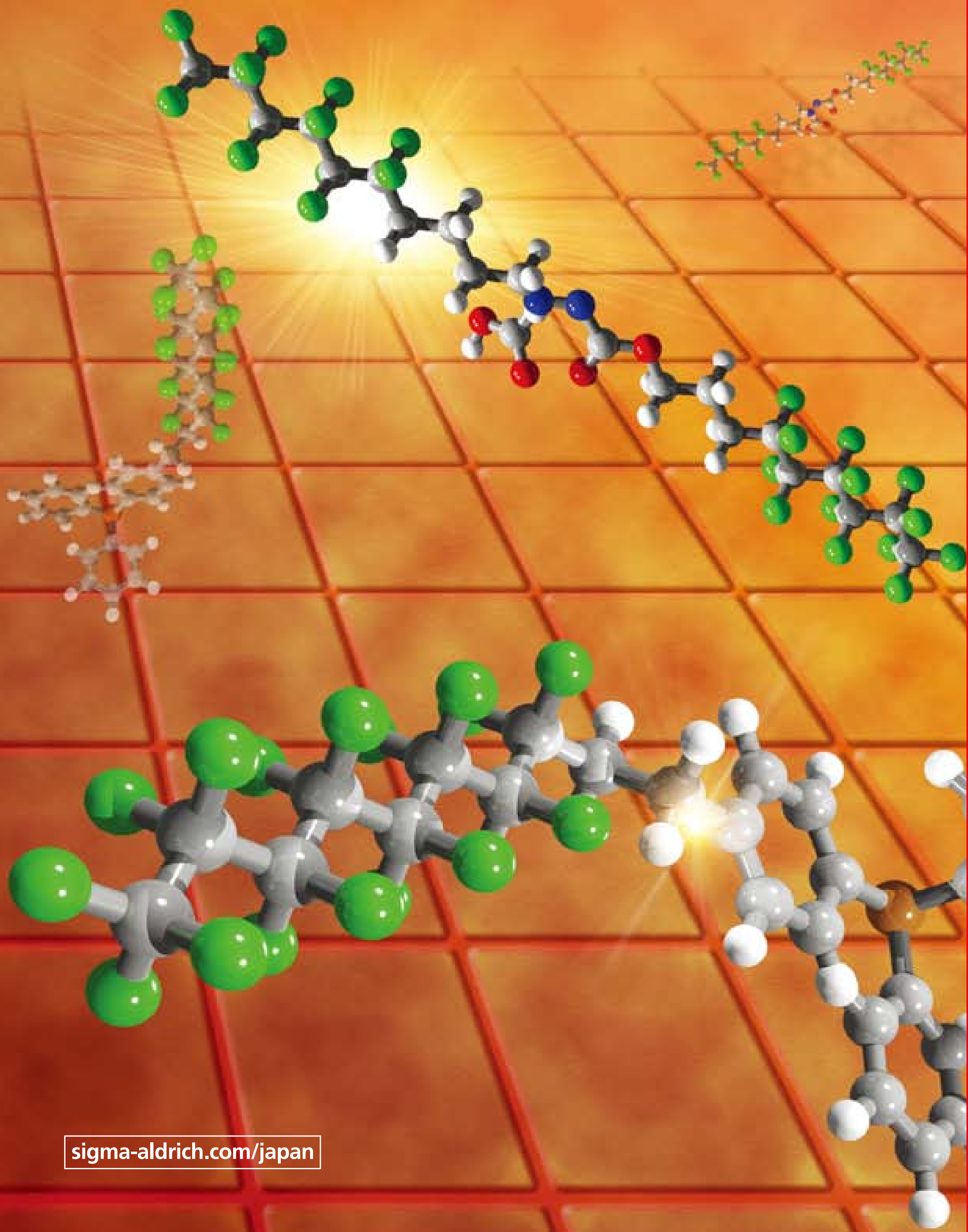
ChemFiles

Vol. 4 No. 9

Fluorous Synthesis and Separation

フルオラス化学
～合成と分離～



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Products for Fluorous Synthesis and Separation
フルオラス化学　～合成と分離～

Introduction

Sigma-Aldrichが新たにFlourous Technologies, Inc. 社の製品をご提供することになりました！その他にも、種々のフルオラス試薬、触媒、リガンド、保護基、スカベンジャー、溶媒、そして固相抽出用吸着剤・カラムなど関連製品を幅広く扱っております。フルオラス化学研究用の製品はSigma-Aldrichにお任せください。

フルオラス化学の手法は、グリーンケミストリーの観点からのプロセス開発と基礎化学研究のいずれにも応用可能です。精製効率が良いので生産性が向上します。反応混合物から目的物質を分離精製する方法としてレジン試薬同様に、一般性があり幅広い反応系に応用でき、しかも確実にかつ選択的に作用しますが、フルオラス化学にはさらに、反応速度を増大、反応の進行状況を追跡可能、従来の液相反応を応用可能といったレジン試薬にはない利点があり、ほとんどの液相有機反応に応用できます。また、フルオラスタグ法は固相合成を用いることのできない均一系の化学反応にも利用でき、従来から慣れ親しんだ実験法（液相反応、液相－液相抽出、固相－液相抽出）をそのまま活用できる点が魅力です。このように、多岐にわたる化学反応の従来法が踏襲できる一方、フルオラス化学は、自動合成、超臨界CO_2、マイクロ波合成などの新しい合成技術とも併用できます。

フルオラス化学に関するこれまでの報告は、フルオラス2相触媒、フルオラス3相反応、フルオラス試薬、フルオラス基質、フルオラス混合合成（Fluorous Mixture Synthesis）の5つの分野に大別されます。詳細は下記の参考文献をご覧ください。多くの新技術は液相パラレル合成によるコンビナトリアルライブラリーの合成に適しており、フルオラスタグの長さと性質、反応条件、さらに分離方法によって、さまざまな手法が見出されています。

一般に、長鎖Rf基を有するフルオラスタグ化試薬（heavy fluorous compounds）は、液相－液相抽出またはフルオラス固相抽出を併用する創薬化学合成、及び多種多様な有機分子のタグに適しています。Rf基が短いフルオラスタグ化試薬（light fluorous compounds）は、フルオラスクロマトグラフィーやフルオラス混合合成（Fluorous Mixture Synthesis）に有用です。小さいフルオラスタグの方が分子量が低く有機溶媒への溶解性が高いので、固相反応であれ液相反応であれ分離段階でのみフルオラス相を必要とし、反応にはフルオラス溶媒を必要としない場合もあります。Sigma-Aldrichは、heavy/lightのいずれのフルオラスタグ化試薬もご提供しております。

General Reviews on Fluorous Techniques:

[1] Gladysz, J. A.; Curran, D. P. *Tetrahedron* **2002**, *58*, 3823–3825; [2] Tzschucke, C.C.; Markert, C.; Bannwarth, W.; Roller, S.; Hebel, A.; Haag, R. *Angew. Chem. Int. Ed. Engl.* **2002**, *41*, 3964–4000; [3] Curran, D. P.; Hadida, S.; Studer, A.; He, M.; Kim, S.-Y.; Luo, Z.; Larhed, M.; Hallberg, A.; Linclau, B. In *Combinatorial Chemistry: A Practical Approach*; H. Fenniri, Ed.; Oxford Univ Press: Oxford, 2000; Vol. 2, pp 327–352; [4] Curran, D. P. In *Stimulating Concepts in Chemistry*, Wiley-VCH, 2000, 25–37; [5] de Wolf, E.; Van Kloten, G.; Deelman, B.-J. *Chem. Soc. Review* **1999**, *28*, 37–41; [6] Betzemeier, B.; Knochel, P. *Top. Curr. Chem.* **1999**, *206*, 61–78; [7] Fish, R. H. *Chem. Eur. J.* **1999**, *5*, 1677–1680; [8] Maul, J. J.; Ostrowski, P. J.; Ublacker, G. A.; Linclau, B.; Curran, D. P. In *Topics in Current Chemistry, Modern Solvents in Organic Synthesis*; P. Knochel, Ed.; Springer-Verlag: Berlin, 1999; Vol. 206, pp 80–104; [9] Curran, D. P. *Angew. Chem. Int. Ed. Engl.* **1998**, *37*, 1175–1196; [10] Horváth, I. T. *Acc. Chem. Res.* **1998**, *31*, 641–650.

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Email：sialjpts@sial.com

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1. Rf-tagged Protecting Groups—Temporary, Cleavable, Fluorous Tags

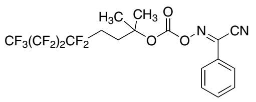
1.1 F-Boc-ON

F-Boc-ON is the fluorous equivalent of 2-(tert-butoxycarbonyloxyimino)-2-phenylacetonitrile (Boc-ON) used in protecting amino groups in peptide synthesis or other functionalities in multi-step organic synthesis. Protection of the amino group with F-Boc-ON and deprotection are achieved under traditional reaction conditions, with the advantage that products containing the F-Boc group can easily be separated from organic reagents, reactants, or products by performing a quick fluorous solid-phase extraction over Fluoro*Flash*® Silica Gel (see sections 8 and 9).[1,2]

Lit.: [1] Luo, Z. Y.; Williams, J.; Read, R. W.; Curran, D. P. *J. Org. Chem.* **2001**, *66*, 4261; [2] Curran, D. P. *Synlett* **2001**, 1488.

2-[(4,4,5,5,6,6,7,7,7-Nonafluoro-1,1-dimethylheptyloxy)carbonyloxyimino]-2-phenylacetonitrile,[1] ≥97%

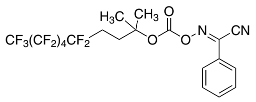
01382
$C_{18}H_{15}F_9N_2O_3$
Mw 478.31



1 g ¥13,300

2-[(4,4,5,5,6,6,7,7,8,8,9,9,9-Tridecafluoro-1,1-dimethylnonyloxy)carbonyloxyimino]-2-phenylacetonitrile,[1] ≥97%

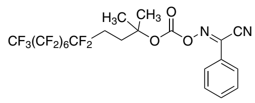
11807
$C_{20}H_{15}F_{13}N_2O_3$
Mw 578.32



1 g ¥13,800
5 g ¥55,000

2-[(4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Heptadecafluoro-1,1-dimethylundecyloxyimino]-2-phenylacetonitrile,[1] ≥97%

55118
$C_{22}H_{15}F_{17}N_2O_3$
Mw 678.34
[350716-42-6]



1 g ¥11,400
5 g ¥45,700

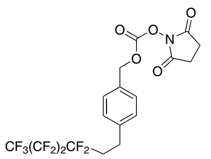
1.2 F-Cbz-OSu

F-Cbz-OSu is the fluorous equivalent of benzyloxycarbonyloxysuccinimide (Cbz-OSu) used in protecting amino groups in peptide synthesis or multi-step organic synthesis. Protection of the amino group with F-Cbz-OSu and subsequent deprotection are achieved with traditional reaction conditions, with the advantage that products containing the F-Cbz group can easily be separated from organic reagents, reactants, or products by performing a quick fluorous solid-phase extraction over Fluoro*Flash*® Silica Gel (see sections 8 and 9).[1,2]

Lit.: [1] Curran, D. P.; Amatore, M.; Guthrie, D.; Campbell, M.; Go, E.; Luo, Z. **2003**, *68*, 4643; [2] Curran, D. P. *Synlett* **2001**, 1488.

N-[4-(3,3,4,4,5,5,6,6,6-Nonafluorohexyl)benzyloxycarbonyloxy] succinimide,[1] ≥95%

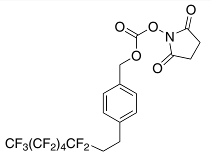
00246
$C_{18}H_{14}F_9NO_5$
Mw 495.29



1 g ¥23,100

N-[4-(3,3,4,4,5,5,6,6,7,7,8,8,8-Tridecafluorooctyl)benzyloxy-carbonyloxy] succinimide,[1] ≥97%

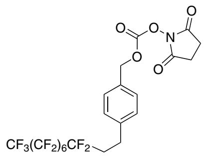
05656
$C_{20}H_{14}F_{13}NO_5$
Mw 595.31
[556050-48-7]



1 g ¥19,900
5 g ¥79,200

N-[4-(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-Heptadecafluoro-decyl)benzyloxycarbonyloxy] succinimide,[1] ≥97%

14944
$C_{22}H_{14}F_{17}NO_5$
Mw 695.32
[556050-49-8]



1 g ¥20,400
5 g ¥81,000

1.3 F-PMB-OH

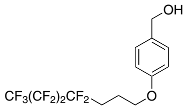
F-PMB-OH is the fluorous equivalent of p-methoxybenzyl alcohol (PMB-OH) used in protecting alcohols in multi-step organic synthesis.

Protection of an alcohol with F-PMB-OH and deprotection are achieved under traditional reaction conditions.[1,2]

Lit.: [1] Curran, D. P.; Furukawa, T. *Org. Lett.* **2002**, 2233; [2] Curran, D. P. *Synlett* **2001**, 1488.

4-(4,4,5,5,6,6,7,7,7-Nonafluoroheptyloxy)benzyl alcohol,[1] ≥97%

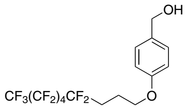
01452
$C_{14}H_{13}F_9O_2$
Mw 384.24



1 g ¥17,000

4-(4,4,5,5,6,6,7,7,8,8,9,9,9-Tridecafluorononyloxy)benzyl alcohol,[1] ≥97%

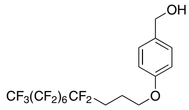
67772
$C_{16}H_{13}F_{13}O_2$
Mw 484.25



1 g ¥22,500
5 g ¥89,600

4-(4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Heptadecafluoro-undecyloxy)benzyl alcohol,[1] ≥97 %

97071
$C_{18}H_{13}F_{17}O_2$
Mw 584.27

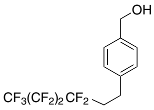


1 g ¥13,400
5 g ¥53,300

1.4 F-Benzyl Alcohol

4-(3,3,4,4,5,5,6,6,6-Nonafluorhexyl)benzyl alcohol,[1] ≥95%

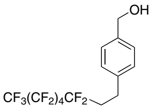
08431
$C_{13}H_{11}F_9O$
Mw 354.21



1 g ¥17,000

4-(3,3,4,4,5,5,6,6,7,7,8,8,8-Tridecafluorooctyl)benzyl alcohol,[1] ≥97%

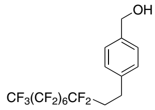
16638
$C_{15}H_{11}F_{13}O$
Mw 454.23
[356055-76-0]



1 g ¥17,000
5 g ¥72,000

4-(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-Heptadecafluorodecyl-benzyl alcohol,[1] ≥98%

19563
$C_{17}H_{11}F_{17}O$
Mw 554.24
[356055-77-1]



1 g ¥13,400
5 g ¥53,600

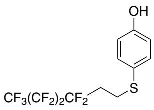
1.5 FluoMar™

FluoMar™ is the fluorous equivalent of the Marshall resin used frequently as a carbonate and carbamate linker in solid-phase syntheses.[1,2] This reagent can be used as an alternative to the Marshall resin in solution-phase combinatorial and parallel synthesis. Tagging of substrates is achieved with traditional reaction conditions, with the advantage that products or intermediates containing the tag (FluoMar™) group can easily be separated from organic reagents, reactants or products by performing a quick solid-phase extraction over Fluoro*Flash*® silica gel.[3]

Lit.: [1] Chen, C. H.-T.; Zhang, W. *Org. Lett.* **2003**, *5*, 1015; [2] Marshall, D. L.; Liener, I. E. *J. Org. Chem.* **1970**, *35*, 867; [3] Curran, D. P. *Synlett* **2001**, 1488.

4-(3,3,4,4,5,5,6,6,6-Nonafluorohexylthio)phenol,[1] ≥90 %

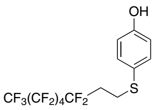
43849
$C_{12}H_9F_9OS$
Mw 372.25



1 g ¥5,700

4-(3,3,4,4,5,5,6,6,7,7,8,8,8-Tridecafluorooctylthio)phenol,[1] ≥97%

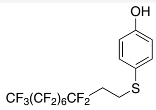
43893
$C_{14}H_9F_{13}OS$
Mw 472.26



1 g ¥6,300
5 g ¥25,300

4-(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-Heptadecafluorodecyl-thio)phenol,[1] ≥97%

40829
$C_{16}H_9F_{17}OS$
Mw 572.28
[142623-70-9]



1 g ¥6,000
5 g ¥24,000

[1] The Rf-tagged protecting groups are products of Fluorous Technologies, Inc. Use of these compounds may be protected by U.S. patents 6,156,896; 5,859,247; 5,777,121 and 6,673,539.

2. Rf-tagged Solution-Phase Scavengers

Rf-tagged Solution-Phase Scavengers are fluorous-modified versions of familiar electrophilic and nucleophilic scavengers. Perfluoroalkyl tags are introduced to the scavenging moiety and subsequently facilitate rapid separation of the scavenged species from target products using fluorous, packed SPE cartridges, chromatography, or liquid extraction. The strategy is the same as for conventional resin or silica-supported scavengers, except that fluorous-supported scavengers allow to work in homogenous solutions.

Advantages over solid-phase scavengers include:
- Rapid quenching without large excess of scavenging reagent
- Scalability
- Separation by either solid-phase or liquid-liquid extraction
- Chemically inert and thermally stable tags

2.1 Rf-tagged Nucleophilic Scavengers

2.1.1 F-Thioles

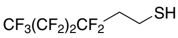
Thiols are good nucleophiles that have been used as covalent scavengers to rid product mixtures of excess halides and other electrophiles. F-Thiol is the solution-phase equivalent of polymer bound thiol scavengers. It has the advantage of reacting faster than its polymer counterpart.[1] F-Thiol-quenched derivatives can be easily separated from the organic product by performing a quick solid-phase extraction over Fluoro*Flash*® silica gel.[2,3]

F-Thiols can also serve as fluorous tags in parallel synthesis.[4] Tagging is achieved by nucleophilic substitution of a halide by the thiol. Detagging is achieved by oxidation of the sulfide to a sulfone followed by nucleophilic substitution by another nucleophile.

Lit.: [1] Zhang, W.; Curran, D. P.; Chen, H.-T. *Tetrahedron* **2002**, 3871; [2] Curran, D. P. *Synlett* **2001**, 1488; [3] Lindsley, C. W.; Zhao, Z.; Leister, W. H.; Strauss, K. A. *Tetrahedron Lett.* **2002**, *43*, 6319; [4] Zhang, W. *Org. Lett.* **2003**, *5(7)*, 1011.

3,3,4,4,5,5,6,6,6-Nonafluoro-1-hexanethiol,[1] ≥99%

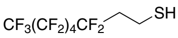
02507
$C_6H_5F_9S$
Mw 280.15
[34451-25-7]



1 g ¥11,800

3,3,4,4,5,5,6,6,7,7,8,8,8-Tridecafluoro-1-octanethiol,[1] ≥99%

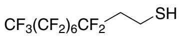
02527
$C_8H_5F_{13}S$
Mw 380.17
[34451-26-8]



1 g ¥4,400
5 g ¥19,000

3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-Heptadecafluoro-1-decanethiol,[1] ≥99%

08686
$C_{10}H_5F_{17}S$
Mw 480.18
[34143-74-3]

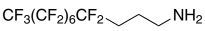


1 g ¥3,900
5 g ¥15,500

2.1.2 F-Amines

4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Heptadecafluoro-undecylamine,[1] ≥97%

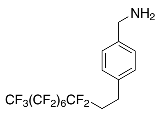
04636
$C_{11}H_8F_{17}N$
Mw 477.16
[139175-50-1]



1 g ¥12,000
5 g ¥48,000

4-(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-Heptadecafluorodecyl) benzylamine,[1] ≥97%

07856
$C_{17}H_{12}F_{17}N$
Mw 553.26
[609816-23-1]



1 g ¥25,700
5 g ¥100,100

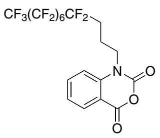
[1] The Rf-tagged nucleophilic scavengers are products of Fluorous Technologies, Inc. Use of these compounds may be protected by U.S. patents 6,156,896; 5,859,247; 5,777,121 and 6,673,539.

2.2 Rf-tagged Electrophilic Scavengers

2.2.1 F-Isatoic Anhydride

1-(4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Heptadecafluoroundecyl)-3,1-benzoxazine-2,4(1H)-dione,[1] ≥97%

07172
$C_{19}H_{10}F_{17}NO_3$
Mw 623.26
[544418-04-4]

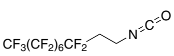


1 g ¥16,100
5 g ¥63,100

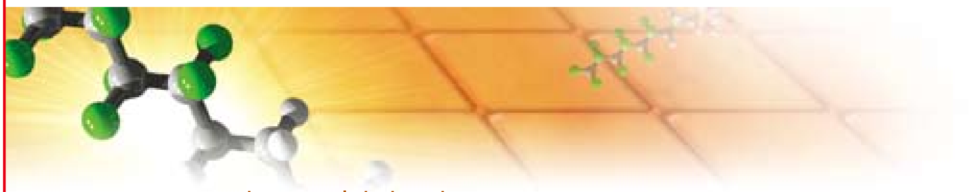
2.2.2 F-Ethyl Isocyanate

3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-Heptadecafluorodecyl isocyanate,[1] ≥97%

18486
$C_{11}H_4F_{17}NO$
Mw 489.13
[142010-50-2]



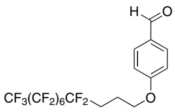
1 g ¥13,600
5 g ¥58,000



2.2.3 F-Oxybenzaldehyde

4-(4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Heptadecafluoro-undecyloxy) benzaldehyde,[1] ≥97%

38456
$C_{18}H_{11}F_{17}O_2$
Mw 582.25
[494798-73-1]



1 g ¥11,700
5 g ¥46,300

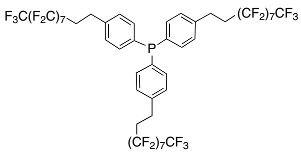
[1]Rf-tagged electrophilic scavengers are products of Fluorous Technologies, Inc. Use of this compound may be protected by U.S. patents 6,156,896; 5,859,247; 5,777,121 and 6,673,539

3. Rf-tagged Ligands and Organometallic Complexes

3.1 F-Triphenylphosphines

Tris[4-(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heptadecafluoro-decyl)phenyl]phosphine, ≥97%

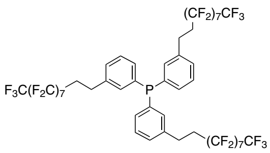
84928
$C_{48}H_{24}F_{51}P$
Mw 1600.60
[325459-92-5]



1 g ¥34,200
5 g ¥136,600

Tris[3-(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heptadecafluoro-decyl)phenyl]phosphine, ≥95%

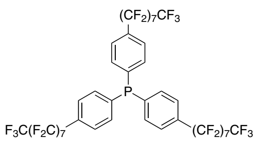
83934
$C_{48}H_{24}F_{51}P$
Mw 1600.60
[342889-38-7]



1 g ¥34,200
5 g ¥136,600

Tris[4-(heptadecafluorooctyl)phenyl]phosphine,[1] ≥85%

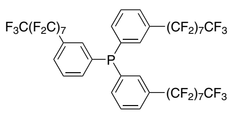
07086
$C_{42}H_{12}F_{51}P$
Mw 1516.44
[284472-92-0]



1 g ¥21,000
5 g ¥83,600

Tris[3-(heptadecafluorooctyl)phenyl]phosphine, ≥95%

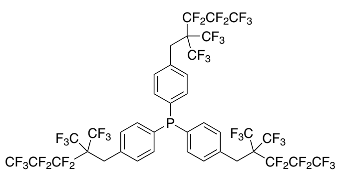
49822
$C_{42}H_{12}F_{51}P$
Mw 1516.44
[325459-91-4]



1 g ¥46,000
5 g ¥166,500

Tris[4-(3,3,4,4,5,5,5-heptafluoro-2,2-bis(trifluoromethyl) pentyl)phenyl] phosphine,[1] ≥90%

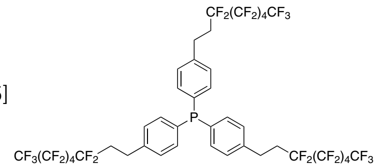
67301
$C_{39}H_{18}F_{39}P$
Mw 1258.47
[322647-82-5]



1 g ¥25,000
5 g ¥99,000

Tris[4-(3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctyl)phenyl]-phosphine,[1] ≥95%

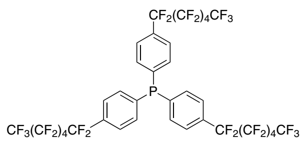
49317
$C_{42}H_{24}F_{39}P$
Mw 1300.55
[219985-31-6]



1 g ¥20,000
5 g ¥95,800

Tris[4-(tridecafluorohexyl)phenyl]phosphine,[1] ≥90%

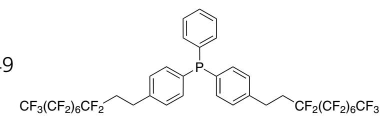
12118
$C_{36}H_{12}F_{39}P$
Mw 1216.39
[193197-68-1]



1 g ¥21,200
5 g ¥83,600

Bis[4-(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heptadecafluoro-decyl)phenyl]phenylphosphine,[1] ≥97%

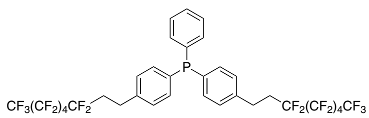
16367
$C_{38}H_{21}F_{34}P$
Mw 1154.49



1 g ¥24,400
5 g ¥97,000

Bis[4-(3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctyl)phenyl]-phenylphosphine,[1] ≥90%

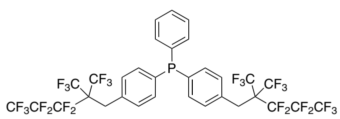
19971
$C_{34}H_{21}F_{26}P$
Mw 954.46
[290827-94-0]



1 g ¥21,000
5 g ¥89,200

Bis[4-(3,3,4,4,5,5,5-heptafluoro-2,2-bis(trifluoromethyl)-pentyl)phenyl]phenylphosphine,[1] ≥90%

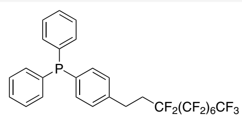
50476
$C_{32}H_{17}F_{26}P$
Mw 926.41
[322647-83-6]



1 g ¥22,800
5 g ¥91,000

[4-(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-Heptadecafluoro-decyl)phenyl] diphenylphosphine,[1] ≥99%

07026
$C_{28}H_{18}F_{17}P$
Mw 708.39
[462996-04-9]



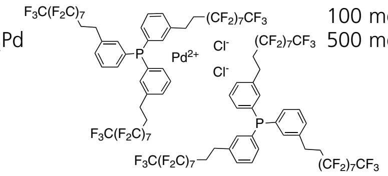
1 g ¥23,900
5 g ¥95,800

[1] Products of Fluorous Technologies, Inc. Use of these compounds may be protected by U.S. patents 6,156,896; 5,859,247; 5,777,121 and 6,673,539.

3.2 F-Bis(triphenylphosphine)-Pd(II) dichloride Complexes

Bis-[tris(3-(1*H*,1*H*,2*H*,2*H*-perfluorodecyl)-phenyl)-phosphine] palladium(II) dichloride, ≥90%

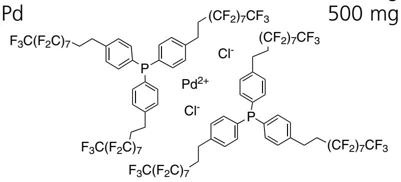
93521
$C_{96}H_{48}Cl_2F_{102}P_2Pd$
Mw 3378.55



100 mg ¥13,200
500 mg ¥51,300

Bis-[tris(4-(1*H*,1*H*,2*H*,2*H*-perfluorodecyl)-phenyl)-phosphine] palladium(II) dichloride, ≥80%

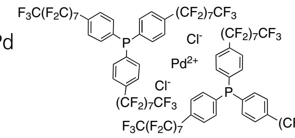
95447
$C_{96}H_{48}Cl_2F_{102}P_2Pd$
Mw 3378.55



100 mg ¥13,200
500 mg ¥51,300

Bis-[tris-(3-(heptadecafluorooctyl)-phenyl)-phosphine] palladium(II) dichloride, ≥95%

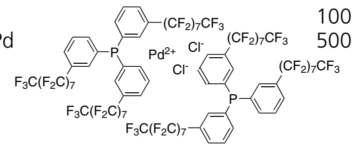
95421
$C_{84}H_{24}Cl_2F_{102}P_2Pd$
Mw 3210.20



100 mg ¥11,400
500 mg ¥45,500

Bis-[tris-(4-(heptadecafluorooctyl)-phenyl)-phosphine] palladium(II) dichloride, ≥95%

88508
$C_{84}H_{24}Cl_2F_{102}P_2Pd$
Mw 3210.20



100 mg ¥11,400
500 mg ¥45,500

3.3 Fluorous Biphase Catalysis, Kit I: C–C-Coupling

The Fluorous Biphase Catalysis Kit for C–C-Couplings contains perfluoro-tagged Pd-catalysts and solvents for up to 10 different catalytic reactions under fluorous biphasic conditions. To get acquainted with this innovative FBC technology our kit provides detailed descriptions of procedures for two C–C-coupling reactions (Suzuki and Stille coupling),[1,2] all substrates and reagents necessary for these two model reactions, and eight additional preparations. Analytical methods and spectra are also given as references. FBC technology allows the catalyst to be easily recovered and to be used for further syntheses.

Lit.: [1] Schneider, S.; Bannwarth, W. *Helv. Chem. Acta* **2001**, *84*, 1. [2] Schneider, S.; Bannwarth, W. *Angew. Chem., Int. Ed. Engl.* **2000**, *39*, 4142.

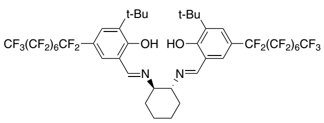
Fluorous Biphase Catalysis, Kit I: C–C-Coupling

67456 1 kit ¥71,900

3.4 F-Salen Ligand and F-Co-Salen Complex

N,N'-Bis[3-tert-butyl-5-(heptadecafluorooctyl)salicylidene)-trans-1,2-cyclohexanediamine,[1] ≥90%

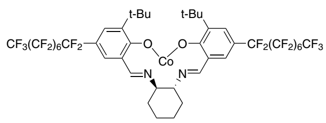
04167
$C_{44}H_{36}F_{34}N_2O_2$
Mw 1270.71



1 g ¥30,900
5 g ¥121,900

N,N'-Bis[3-tert.-butyl-5-(heptadecafluorooctyl)salicylidene)-trans-1,2-cyclohexanediamine-cobalt(II),[1] techn.

04168
$C_{44}H_{34}CoF_{34}N_2O_2$
Mw 1327.63



1 g ¥36,400
5 g ¥143,900

[1]Products of Fluorous Technologies, Inc. Use of this compound may be protected by U.S. patents 6,156,896; 5,859,247; 5,777,121 and 6,673,539

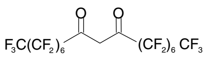
3.5 Symmetrical Rf-tagged 1,3-Diketone Ligand

Triacontafluoro-8,10-heptadecanedione has been described as an symmetrical perfluorinated ligand for transition metal catalyzed oxidations in perfluorinated solvents.[1]

Lit.: [1] Klement, I.; Lütjens, H.; Knochel, P. *Angew. Chemie, Int. Ed. Engl.* **1997**, *26*, 1454

Triacontafluoro-8,10-heptadecanedione, ≥99.0%

03393
$C_{17}H_2F_{30}O_2$
Mw 808.15
[36554-97-9]



100 mg ¥31,600
500 mg ¥124,800

4. Rf-tagged Reagents

4.1 F-DIAD

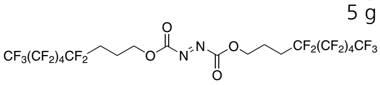
In a typical Mitsunobu reaction between an acidic pronucleophile and an alcohol promoted by diisopropylazodicarboxylate (DIAD) and triphenylphosphine, the desired product is contaminated by reagent derived by-products, phosphine oxide, and hydrazide. F-triphenylphosphine (# **07026**, see section 3.1) and F-DIAD (# **68333**, below) can be used in traditional solution-phase Mitsunobu chemistry.[1] The by-products of the reaction are easily separated from the desired product by performing a quick fluorous solid-phase extraction over Fluoro*Flash*® silica gel.[2] The original fluorous azodicarboxylate, F-DEAD, worked well for carboxylic acid and phthalimide pronucleophiles, but generally less well for phenols and other higher pKa acids. By employing F-DIAD reagent, it is now possible to conduct fluorous Mitsunobu reactions with almost all classes of pronucleopohiles including phenols.

Other fluorous phosphine analogs (see section 3.1) with increased number of fluorine atoms may be used. Those with the highest fluorine content are suitable for reactions and separations under biphasic conditions. The phosphine oxide residue may be removed by liquid-liquid extraction as an alternative to SPE.

Lit.: [1] Dandapani, S.; Curran, D. P. *Tetrahedron* **2002**, 3855–3864; [2] Curran, D. P. *Synlett* **2001**, 1488.

Bis(4,4,5,5,6,6,7,7,8,8,9,9,9-tridecafluorononyl)azo-dicarboxylate,[1] ≥90%

68333
$C_{20}H_{12}F_{26}N_2O_4$
Mw 838.28
[462996-01-6]

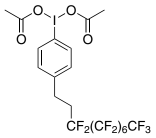


1 g ¥19,900
5 g ¥79,800

4.2 F-DAIB

1-(Diacetoxyiodo)-4-(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heptadecafluorodecyl)benzene,[1] ≥90%

16429
$C_{20}H_{14}F_{17}IO_4$
Mw 768.20



1 g ¥31,500
5 g ¥124,800

[1] The Rf-tagged reagents are products of Fluorous Technologies, Inc. Use of these compounds may be protected by U.S. patents 6,156,896; 5,859,247; 5,777,121 and 6,673,539.

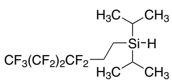
5. Rf-tagged Silanes

F-Silanes are fluorous equivalents to a TIPS group and exhibit properties similar to most silicon protecting groups. Silanes have been used as fluorous tags in both parallel and fluorous mixture synthesis (FMS).[1,2] Tagging of an alcohol is accomplished by in situ activation of the F-Silane to either the bromide or the triflate followed by addition of the alcohol. The tagged molecule can be manipulated over a number of chemical steps before detagging with fluoride. The tagged intermediates are quickly and easily separated after each step from organic reagents, reactants, or products by fluorous solid-phase extraction (F-SPE) over Fluoro*Flash*® Silica Gel.[3]

Lit.: [1] Luo, Z. Y.; Zhang, Q.; Oderaotoshi, Y.; Curran, D. P. *Science* **2001**, *291*, 1766; [2] Zhang, W.; Luo, Z.; Chen, C. H.; Curran, D. P. *J. Am. Chem. Soc.* **2002**, *124*, 10443; [3] Curran, D. P. *Synlett* **2001**, 1488.

Diisopropyl(3,3,4,4,5,5,6,6,6-nonafluorohexyl)silane,[1] ≥95%

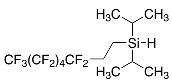
18976
$C_{12}H_{19}F_9Si$
Mw 362.35
[356056-13-8]



1 g ¥15,300

Diisopropyl(3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctyl) silane,[1] ≥95%

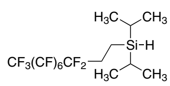
00454
$C_{14}H_{19}F_{13}Si$
Mw 462.37
[356056-14-9]



1 g ¥14,000
5 g ¥55,900

Diisopropyl(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heptadeca-fluorodecyl)silane,[1] ≥95%

04537
$C_{16}H_{19}F_{17}Si$
Mw 562.38
[356056-15-0]



1 g ¥11,600
5 g ¥46,200

[1] The Rf-tagged silanes are products of Fluorous Technologies, Inc. Use of these compounds may be protected by U.S. patents 6,156,896; 5,859,247; 5,777,121 and 6,673,539.

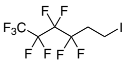
6. Rf-tagged Building Blocks and Precursors

6.1 Rf-tagged Alkyl Iodides

6.1.1 F-Ethyl Iodide

1,1,1,2,2,3,3,4,4-Nonafluoro-6-iodohexane, ≥97%

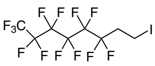
07387
$C_6H_4F_9I$
Mw 373.99
[2043-55-2]



5 g	¥5,300
25 g	¥17,300

1,1,1,2,2,3,3,4,4,5,5,6,6-Tridecafluoro-8-iodooctane, ≥95.0%

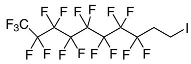
77309
$C_8H_4F_{13}I$
Mw 474.00
[2043-57-4]



5 mL	¥12,000
25 mL	¥45,000

1,1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8-Heptadecafluoro-10-iodododecane, >95.0%

80219
$C_{10}H_4F_{17}I$
Mw 574.02
[2043-53-0]

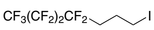


5 g	¥3,600
25 g	¥14,000
100 g	¥45,000

6.1.2 F-Propyl Iodide

4,4,5,5,6,6,7,7,7-Nonafluoroheptyl iodide,[1] ≥97%

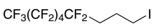
52961
$C_7H_6F_9I$
Mw 388.02
[183547-74-2]



1 g	¥7,800
5 g	¥31,000

4,4,5,5,6,6,7,7,8,8,9,9,9-Tridecafluorononyl iodide,[1] ≥97%

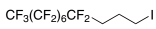
16548
$C_9H_6F_{13}I$
Mw 488.03
[89889-20-3]



1 g	¥8,200
5 g	¥35,000

4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Heptadecafluoro-undecyl iodide,[1] ≥99%

68794
$C_{11}H_6F_{17}I$
Mw 588.04
[200112-75-0]



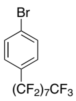
1 g	¥5,000
5 g	¥20,700

[1] The F-propyl-iodides are products of Fluorous Technologies, Inc. Use of these compounds may be protected by U.S. patents 6,156,896; 5,859,247; 5,777,121 and 6,673,539.

6.2. Rf-tagged Bromobenzene

1-Bromo-4-(heptadecafluorooctyl)benzene, ≥97%

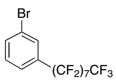
40859
$C_{14}H_4BrF_{17}$
Mw 575.06
[206560-77-2]



1 g	¥11,400
5 g	¥45,500

1-Bromo-3-(heptadecafluorooctyl)benzene, ≥95%

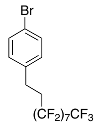
76071
$C_{14}H_4BrF_{17}$
Mw 575.06
[325459-90-3]



1 g	¥11,400
5 g	¥45,500

1-Bromo-4-3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heptadeca-fluorodecyl)benzene, ≥97%

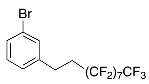
93037
$C_{16}H_8BrF_{17}$
Mw 603.11
[195324-88-0]



1 g	¥10,900
5 g	¥43,600

1-Bromo-3-(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heptadeca-fluorodecyl)benzene, ≥99%

87912
$C_{16}H_8BrF_{17}$
Mw 603.11
[340157-97-3]

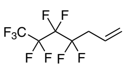


1 g	¥13,900
5 g	¥55,700

6.3 Rf-tagged Ethylene

1H,1H,2H-Perfluoro-1-hexene

ZONYL®PFBE fluorotelomer intermediate
42,150-2
$C_6H_3F_9$
Mw 246.08
[19430-93-4]

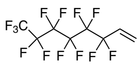


25 mL	¥8,900
100 mL	¥24,600

DuPont product, ®Registered trademark of E.I. du Pont de Nemours & Co., Inc.

1H,1H,2H-Perfluoro-1-octene,≥99.0%

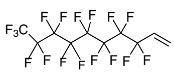
37,056-8
$C_8H_3F_{13}$
Mw 346.09
[25291-17-2]



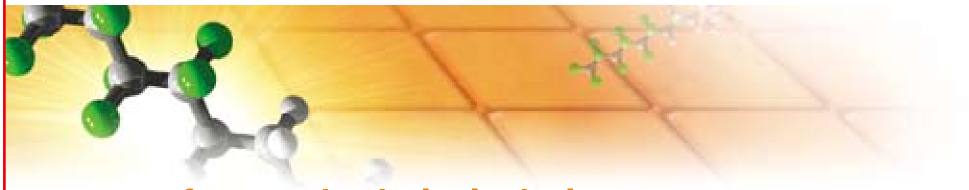
5 g	¥5,500
25 g	¥18,300

3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-Heptadecafluoro-1-decene, 99 %

37,057-6
$C_{10}H_3F_{17}$
Mw 446.11
[21652-58-4]

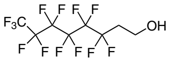


5 g	¥6,900
25 g	¥20,000



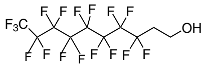
6.4 Rf-tagged Ethyl Alcohol

1H,1H,2H,2H-Perfluoro-1-octanol, ≥97.0%



77278	5 mL	¥9,100
$C_8H_5F_{13}O$	25 mL	¥32,000
Mw 364.11		
[647-42-7]		

1H,1H,2H,2H-Perfluoro-1-decanol, ≥90 %

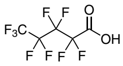


77263	2.5 g	¥2,500
$C_{10}H_5F_{17}O$	10 g	¥8,000
Mw 464.12		
[678-39-7]		

6.5 Rf-tagged Carboxylic Acids and Acid Chlorides

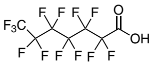
Nonafluorovaleric acid, ≥94 %

Perfluoropentanoic acid



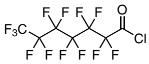
77285	5 mL	¥4,800
$C_5HF_9O_2$	25 mL	¥17,500
Mw 264.05		
[2706-90-3]		

Tridecafluoroheptanoic acid, ≥95.0 %



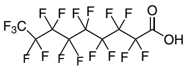
77271	5 mL	¥6,300
$C_7HF_{13}O_2$	25 mL	¥22,500
Mw 364.06		
[375-85-9]		

Tridecafluoroheptanoyl chloride, ≥97.0 %



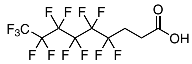
67162	1 g	¥7,100
$C_7ClF_{13}O$	5 g	¥27,300
Mw 382.51		
[52447-22-0]		

Heptadecafluorononanoic acid, ≥95.0%



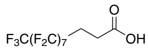
77284	5 g	¥2,900
$C_9HF_{17}O_2$	25 g	¥10,000
Mw 464.08		
[375-95-1]		

4,4,5,5,6,6,7,7,8,8,9,9,9-Tridecafluorononanoic acid, ≥97.0 %



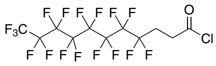
30313	1 g	¥13,200
$C_9H_5F_{13}O_2$	5 g	¥50,600
Mw 392.11		
[27854-30-4]		

4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Heptadecafluoroundecanoic acid, ≥95.0%



05611	1 g	¥26,600
$C_{11}H_5F_{17}O_2$	5 g	¥106,300
Mw 492.13		
[34598-33-9]		

4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Heptadecafluoroundecanoyl chloride, ≥97.0%



71941	1 g	¥25,000
$C_{11}H_4ClF_{17}O$	5 g	¥100,200
Mw 510.57		
[89373-67-1]		

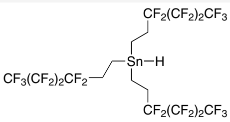
7. Rf-tagged Tin Compounds

Fluorous tagged tin reagents perform the same chemistry as conventional compounds, while also featuring facile purification through fluorous methods.[1-4] The use of fluorous tin hydride in reductive radical cyclizations illustrates many of the features of this branch of fluorous chemistry. In general, the substrate and the product are organic molecules and one of the other reaction components (in this case, the tin hydride) is fluorous. The fluorous component can be used either catalytically or stoichiometrically and the reaction and separation stages are decoupled. After standard reactions, members of the tin hydride family with more fluorines can be separated either by liquid-liquid extraction or by solid liquid extraction, while the solid-liquid extraction is preferred for members with fewer fluorines.

Lit.: [1] Curran, D. P.; Hadida, S. *J. Am. Chem. Soc.* **1996**, *118*, 2531; [2] Curran, D. P.; Hadida, S., Kim, S.-Y.; Luo, Z. *J. Am. Chem. Soc.* **1999**, *121*, 6607; [3] Curran, D. P.; Hadida, S.; Mu He, M. *J. Org. Chem.* **1997**, *62*, 6714; [4] Bucher, B.; Curran, D. P. *Tetrahedron Letters* **2000**, *41*, 9617.

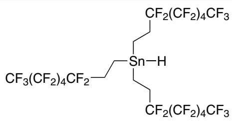
7.1 F-Tin Hydride

Tris(3,3,4,4,5,5,6,6,6-nonafluorohexyl)tin hydride,[2] ~90%



06694	1 g	¥24,000
$C_{18}H_{13}F_{27}Sn$	5 g	¥95,000
Mw 860.96		
[240497-26-1]		

Tris(3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctyl)tin hydride,[2] ~90%



18587	1 g	¥26,000
$C_{24}H_{13}F_{39}Sn$	5 g	¥109,200
Mw 1161.01		
[175354-32-2]		

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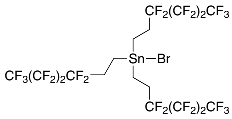
Email：sialjpts@sial.com

7.2 F-Tin Bromide

Tris(3,3,4,4,5,5,6,6,6-nonafluorohexyl)tin bromide,[2] ~ 90%

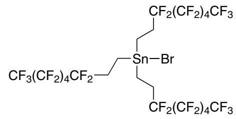
16445
$C_{18}H_{12}BrF_{27}Sn$
Mw 939.86
[240497-37-4]



1 g ¥22,100
5 g ¥88,600

Tris(3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctyl)tin bromide,[2] ≥95%

42710
$C_{24}H_{12}BrF_{39}Sn$
Mw 1239.90
[175354-31-1]

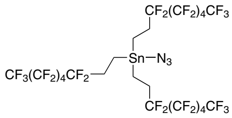


1 g ¥23,200
5 g ¥92,700

7.3 F-Tin Azide

Tris(3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctyl)tin azide,[2] ≥95%

40842
$C_{24}H_{12}F_{39}N_3Sn$
Mw 1202.02
[201740-73-0]

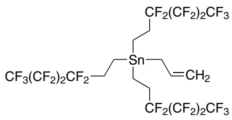


1 g ¥24,100
5 g ¥96,300

7.4 F-Allyl Tin

Allyltris(3,3,4,4,5,5,6,6,6-nonafluorohexyl)stannane,[2] ~90%

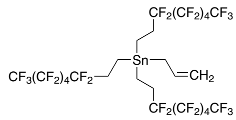
43916
$C_{21}H_{17}F_{27}Sn$
Mw 901.03
[215186-99-5]



1 g ¥26,100
5 g ¥103,000

Allyltris(3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctyl) stannane,[2] ~90%

54114
$C_{27}H_{17}F_{39}Sn$
Mw 1201.07
[192212-66-1]

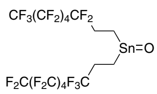


1 g ¥28,800
5 g ¥114,000

7.5 F-Tin Oxide

Bis(3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctyl)tin oxide,[1] ~90%

43482
$C_{16}H_8F_{26}OSn$
Mw 828.90
[324063-66-3]



1 g ¥8,400
5 g ¥35,600

[1] Product of Fluorous Technologies, Inc. Use of this compound may be protected by U.S. patents 6,156,896; 5,859,247; 5,777,121 and 6,673,539.

[2] Products of Fluorous Technologies, Inc. Protected by U.S. patents 6,156,896; 5,859,247; and 5,777,127.

8. Fluorous Separation Media

8.1 Rf-Silica Gel

Fluoro*Flash*® Silica Gel[1], particle size ~40 µm

08965	100 g	¥89,400

8.2 Rf-Silica Gel TLC Plates

Fluoro*Flash*® TLC Plates, with F_{254} indicator,[1] dimension 5 cm x 10 cm

16888	10 ea	¥30,000

[1] The fluorous separation media are products of Fluorous Technologies, Inc. Use of these compounds may be protected by U.S. patents 6,156,896; 5,859,247; 5,777,121 and 6,673,539.

Fluoro*Flash*® is a trademark of Fluorous Technologies, Inc.

9.　Fluoro*Flash*® SPE カラム

フルオラス固相抽出法（F-SPE）は、フルオラス基を有する試薬、保護基、タグやスカベンジャーを反応混合物から簡単に分離する方法です。フルオラス分子はパーフルオロアルキル基が結合したシリカゲルに吸着され非フルオラス分子は極性に無関係に溶出するという性質を利用して、この特殊なシリカゲルを充填したSPEカラムFluoro*Flash*®により、含フッ素分子が混合物から2段階で簡単に分離できます。すなわち、フルオラス化合物を含む反応混合物をF-SPEカラムに注入しフッ素親和性の低い溶媒を流すと、非フルオラス化合物がまず溶出します。その後、メタノール・アセトン・THFなどの有機溶媒を流すとフルオラス化合物が得られます。フルオラスSPEの場合、試料注入量はフルオラスシリカゲルの5-15%が一般的です。また、アセトンやTHFで洗浄することにより、カラムは最大10回まで再利用できます。2種類の溶媒のみを使用するために、複数のフラクションを分取するのには適しませんが、分離能が大きいために大量の試料を精製することができます。この分離法は再生が容易でかつ置換基に左右されないため自動化に最適です。

Lit. : [1] Curran, D. P. *Synlett* **2001**, 1488; [2] Dandapani, S.; Curran, D. P. *Tetrahedron* **2002**, *58*, 3855; [3] Luo, Z.; Williams, J.; Read, R. W.; Curran, D. P. *J. Org. Chem.* **2001**, 4261; [4] Zhang, W. *Org. Lett.* **2003**, *5*, 2555; [5] Zhang, W.; Curran, D. P.; Chen, C. *Tetrahedron* **2002**, *58*, 3871. Lindsley, C. W.; Zhao, Z.; Leister, W. *Tetrahedron Lett.* **2002**, *43*, 4225; [6] Zhang, W.; Luo, Z.; Christine, C.; Curran D. P. *J. Am. Chem. Soc.* **2002**, 124.

2 grams Fluoro*Flash*® Silica Gel/8 cc tube

14196 particle size (Silica Gel) ~40 µm	20 ea	¥67,000

Fluoro*Flash*® SPE Cartridges[1]
5 grams Fluoro*Flash*® Silica Gel/10 cc tube

00866 particle size (Silica Gel) ~40 µm	10 ea	¥75,000

Fluoro*Flash*® SPE Cartridges[1]
10 grams Fluoro*Flash*® Silica Gel/60 cc tube

08967 particle size (Silica Gel) ~40 µm	5 ea	¥75,000

Fluoro*Flash*® SPE Cartridges[1]

Fluoro*Flash*® SPE Cartridges[1]
20 grams Fluoro*Flash*® Silica Gel/60 cc tube

08966 particle size (Silica Gel) ~40 µm	2 ea	¥88,600

Fluoro*Flash*® SPE Cartridges[1]
20 grams Fluoro*Flash*® Silica Gel/60 cc tube

06961 particle size (Silica Gel) ~40 µm	5 ea	¥121,800

[1] Fluoro*Flash*® SPE Cartridges are products of Fluorous Technologies, Inc. Use of these products may be protected by U.S. patents 6,156,896; 5,859,247; 5,777,121 and 6,673,539.

フルオラス固相抽出
Fluorous Separation Using Solid-Phase Extraction

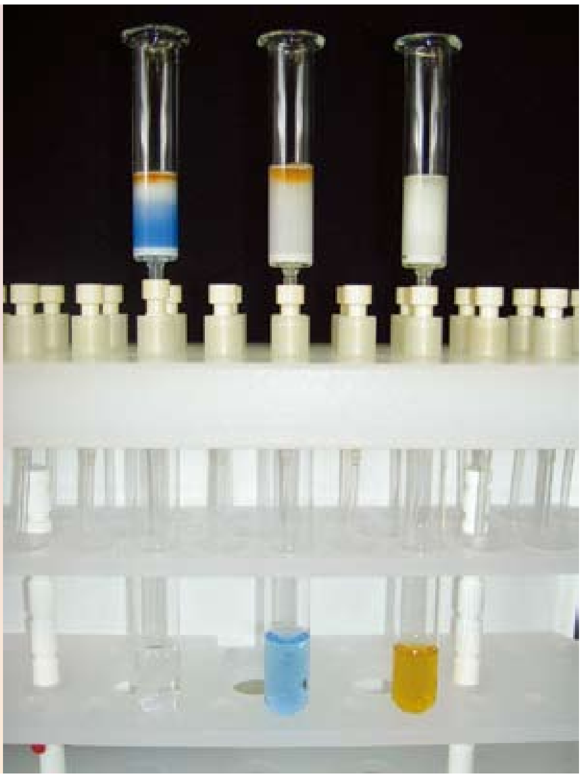
フルオラス固相抽出法を用いれば、2種の溶媒を流すだけでRf基を有する物質を簡単に分離精製できます。
Rf基で修飾した色素（オレンジ色）とRf基を持たない色素（青色）の混合物をFluoro*Flash*®に注入した時の様子を右写真に示しました。

★左のカラム：反応混合物を注入した直後のFluoro*Flash*®.

★中央のカラム：オレンジ色のフルオラスタグ化色素はフッ素-フッ素親和性によりカラムに吸着しているが、MeOH：H_2O=80：20などのフッ素親和性の低い溶媒により青色色素は溶出する（fluorophobic wash）

★右のカラム：メタノールなどの親フッ素溶媒でフルオラスタグ化色素が溶出する（fluorophilic wash）

この簡単な操作により、クロマトグラフィーと同等にしかも素早く精製できます。この分離方法は置換基に左右されないため自動化も容易です。



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Email：sialjpts@sial.com

10. Fluorous Reversed-Phase Silica Gel for the Immobilization of Rf-tagged Catalysts

Easy handling of the catalyst together with its straightforward recovery and possible reuse are important recommendations in any catalytic reaction. The immobilization of perfluoro-tagged Pd catalysts on fluorous reversed-phase silica gel and the successful application to Suzuki and Sonogashira couplings have been described.[1] All couplings were performed in organic solvents without using perfluorinated solvents as second liquid phase. The catalysts showed complete conversions and could be recycled without significant decrease of activity. By adding fluorous reversed-phase silica gel to a solution of a perfluoro-tagged diphenylphosphine Pd(II) dichloride (see section 3.2) in diethyl ether and hexafluorobenzene and evaporating the solvent, an immobilized Pd complex is easily recovered as air-stable, free-flowing powder.

Sigma-Aldrich now offers fluorous reversed-phase silica gel developed for the immobilization of perfluoro-tagged catalysts in three different qualities.

Lit.: [1] Tzschucke, C. C.; Markert, C.; Glatz, H.; Bannwarth, W. *Angew. Chem. Int. Ed.* **2002**, *41*, No 23, 4500.

Silica gel 60 C_8-reversed phase perfluorinated

40915
particle size 0.035–0.070 mm

50 g	¥65,800
250 g	¥259,600

Silica gel 60 C_8-reversed phase perfluorinated end-group silanized

18948
particle size 0.035–0.070 mm

50 g	¥77,400
250 g	¥307,600

Silica gel extra wide pore C_8-reversed phase perfluorinated

18387
particle size 0.1–0.3 mm (50–140 mesh ASTM)

10 g	¥38,000
50 g	¥126,500

11. Solvents for Fluorous Synthesis

Following the definition offered by Gladysz and Curran[1] a "Fluorous Medium" is "any phase of a perfluoroalkane, perfluorodialkylether, perfluorotrialkylamine, or similar non-polar species, or any similarly-composed micro-environment within a non-fluorous medium that shares key physical properties with these species." Fluorous solvents are used in liquid-liquid extractions to quickly separate fluorous compounds from organic compounds in a two phase liquid-liquid extraction, or from organic and inorganic (or water soluble organic) compounds in a three-phase liquid-liquid extraction. Such extractions are readily automated, and can be used to quickly partition reaction mixtures into organic, inorganic and fluorous fractions. In many cases, the crude organic products are pure enough to be taken on to the next reaction, and the fluorous products can usually be recycled, if desired. In the best cases, only a single separation is needed. With lower partition coefficients, the organic fraction is washed several times with the fluorous solvent. Thanks to the exceedingly low solubilities of organic compounds in fluorous solvents, the washing process can be conducted repeatedly without extractive loss of the organic product. Liquid-liquid extractive methods are typically used when the desired product is organic and some other reaction component (reactant, reagent, catalyst, scavenged product) is fluorous. Please note that perfluoroarenes are significantly more polar than perfluoroalkanes and preferentially partition into organic media. Therefore they are not fluorous under the definition above. Partially fluorinated solvents like (trifluoromethyl)-benzene (# **12811**, listed on the next page) provide a homogenous liquid medium for the reaction of fluorous and non-fluorous reactants.[2]

Lit.: [1] Gladysz, J. A.; Curran, D. P. *Tetrahedron* **2002**, *58*, 3823. [2] Curran, D. P.; Hadida, S. *J. Am. Chem. Soc.* **1996**,*118*, 2531.

Fluorinert®-fluid PF-5050

Perfluoropentane mixture of isomers
09973
C_5F_{12}
[678-26-2]

100 mL	¥18,400
500 mL	¥72,800

® Registered Trademark of 3M Corp.

Nonafluorobutyl methyl ether, ≥99.0%

HFE-7100
65139
$C_5H_3F_9O$

250 mL	¥10,200
1 L	¥34,000

1,1,1,2,3,4,4,5,5,5-Decafluoropentane,~60 %

94884
$C_5H_2F_{10}$
[138495-42-8]

100 mL	¥15,600
500 mL	¥61,800

Perfluorohexane FC-72, ~85 %

mixture of isomers
77273
C_6F_{14}
[355-42-0]

10 mL	¥2,000
50 mL	¥6,600

Perfluorohexanes 95%

contains perfluorocyclohexane and ~5% perfluoropentane
37,924-7
C_6F_{14}
[355-42-0]

10 mL	¥3,900
50 mL	¥12,800

Perfluoroheptane, ~80%

mixture of isomers
77272
C_7F_{16}
[335-57-9]

10 mL	¥4,600
50 mL	¥17,300

Octadecafluorooctane, 98.0%

35,923-8
C_8F_{18}
[307-34-6]

25 g	¥8,200
100 g	¥30,700

Octadecafluorooctane Fraction, ≥97.0%

Perfluorooctane fraction (~70% Perfluoroctane)
77286

10 mL	¥6,000
50 mL	¥22,300
250 mL	¥88,100

Perfluorononane, 97.0%

Eicosafluorononane
40,641-4
C_9F_{20}
[375-96-2]

5 mL	¥6,700
25 mL	¥23,300

Dodecafluorocyclohexane, 97%

Perfluorocyclohexane
13,393-0
C_6F_{12}
[355-68-0]

5 g	¥23,900

(Trifluoromethyl)undecafluorocyclohexane, ≥95.0%

Perfluoromethylcyclohexane
77280 — 25 mL ¥8,400; 100 mL ¥20,000; 250 mL ¥44,000
C_7F_{14}
[355-02-2]

Hexadecafluoro-1,3-dimethylcyclohexane, ~80%

Perfluoro-1,3-dimethylcyclohexane
77268 — 50 mL ¥8,000
C_8F_{16}
[335-27-3]

Octadecafluorodecahydronaphthalene, ≥95.0%

Perfluorodecalin
77264 — 10 mL ¥5,100; 50 mL ¥12,200; 250 mL ¥49,100
$C_{10}F_{18}$
[306-94-5]

Perfluoro(methyldecalin) 80%

37,243-9 — 25 g ¥10,000; 100 g ¥14,800
$C_{11}F_{20}$
[51294-16-7]

Hexafluorobenzene, ≥99.0%

52510 — 5 mL ¥3,000; 25 mL ¥7,000; 100 mL ¥21,000
C_6F_6
[392-56-3]

Hexafluorobenzene for NMR-spectroscopy, ≥99.5%

52506 — 5 mL ¥8,200; 25 mL ¥25,800
C_6F_6
[392-56-3]

(Trifluoromethyl)-benzene, ≥98.0%

Benzotrifluoride
12811 — 250 mL ¥4,800; 1 L ¥14,700
$C_7H_5F_3$
[98-08-8]

Heptacosafluorotributylamine

Perfluorotributylamine, Fluorinert® FC-43
77299 — 5 mL ¥4,100; 25 mL ¥17,500
$C_{12}F_{27}N$
[311-89-7]
® Registered Trademark of 3M Corp.

Pentadecafluorotriethylamine, 96%

Perfluorotriethylamine
39,715-6 — 5 mL ¥6,800; 25 mL ¥22,300
$C_6F_{15}N$
[359-70-6]

New Ionic Liquids

イオン性液体は世界的に急激に研究が進められ、近年、その成果には目を見張るものがあります。特殊で優れた性質を持つこの新素材は、学術分野でも数多くの新しい応用例が報告されていますが、最近では、スケールアップした工業的利用も検討され始めています。

CYPHOS®

Cytec Industries, Inc社のテトラアルキルホスホニウム塩CYPHOS®シリーズは、熱に非常に安定であり、従来のイオン性液体とは異なる比重や溶解性を持つために、新たな可能性が期待されているイオン性液体です。

製品番号	製品名				d^{20}_4	容量	価格（¥）
89744	**Trihexyltetradecylphosphonium chloride purum**						
	CYPHOS® IL 101	$C_{32}H_{68}ClP$	Mw 519.31	[258864-54-9]	1.070	5 g; 50 g	¥3,300; ¥12,700
96662	**Trihexyltetradecylphosphonium bromide purum**						
	CYPHOS® IL 102	$C_{32}H_{68}BrP$	Mw 563.76	-	0.960	5 g; 50 g	¥3,300; ¥12,700
50826	**Trihexyltetradecylphosphonium decanoate purum**						
	CYPHOS® IL 103	$C_{42}H_{87}O_2P$	Mw 655.11	[465527-65-5]	0.895	5 g; 50 g	¥3,700; ¥12,700
28612	**Trihexyltetradecylphosphonium bis(2,4,4-trimethylpentyl)phosphinate purum**						
	CYPHOS® IL 104	$C_{48}H_{102}O_2P_2$	Mw 773.27	[465527-58-6]	0.895	5 g; 50 g	¥3,700; ¥12,700
56776	**Trihexyltetradecylphosphonium dicyanamide purum**						
	CYPHOS® IL 105	$C_{34}H_{68}N_3P$	Mw 549.90	-	-	5 g; 50 g	¥4,600; ¥23,900
90145	**Triisobutylmethylphosphonium tosylate purum**						
	CYPHOS® IL 106	$C_{20}H_{37}O_3PS$	Mw 388.54	[344774-05-6]	1.075	5 g; 50 g	¥3,300; ¥12,700
50971	**Trihexyltetradecylphosphonium bis(trifluoromethylsulfonyl)amide purum**						
	CYPHOS® IL109	$C_{34}H_{68}F_6NO_4PS_2$	Mw 764.00	[460092-03-9]	-	5 g; 50 g	¥7,300; ¥45,200
40573	**Trihexyltetradecylphosphonium hexafluorophosphate purum**						
	CYPHOS® IL110	$C_{32}H_{68}F_6P_2$	Mw 628.82	[374683-44-0]	-	5 g; 50 g	¥4,600; ¥23,900
15909	**Trihexyltetradecylphosphonium tetrafluoroborate purum**						
	CYPHOS® IL111	$C_{32}H_{68}BF_4P$	Mw 570.66	[374683-55-3]	2.075	5 g; 50 g	¥3,700; ¥12,700

CYPHOS® is a registered trademark of Cytec Industries, Corp.

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Email：sialjpts@sial.com

BASIONICS™

BASF AGとの提携により、イオン性液体　BASIONICS™シリーズをお求め安い価格でご提供できるようになりました。

製品番号	製品名	mp	d^{20}_{4}	容量	価格（¥）
29164	**1-Ethyl-3-methylimidazolium methanesulfonate BASF Quality, >95%** BASIONIC™ ST 35 $C_7H_{14}N_2O_3S$ Mw 206.27 [145022-45-3]	35 °C	1.2470	100 g; 1 kg	¥30,100; ¥83,000
38938	**Methyl-tri-n-butylammonium methylsulfate BASF Quality, >90%** BASIONIC™ ST 62 $C_{14}H_{33}NO_4S$ Mw 311.49 [13106-24-6]	62 °C	-	100 g; 1 kg	¥15,000; ¥41,500
05338	**1-Ethyl-2,3-dimethylimidazolium ethylsulfate BASF Quality, >95%** BASIONIC™ ST 67 $C_9H_{18}N_2O_4S$ Mw 250.32 [516474-08-1]	67 °C	-	100 g; 1 kg	¥20,000; ¥55,300
38899	**1-Butyl-3-methylimidazolium chloride BASF Quality, >95%** BASIONIC™ ST 70 $C_8H_{15}ClN_2$ Mw 174.67 [79917-90-1]	70 °C	-	100 g; 1 kg	¥20,000; ¥55,300
30881	**1-Butyl-3-methylimidazolium methansulfonate BASF Quality, >95%** BASIONIC™ ST 78 $C_9H_{18}N_2O_3S$ Mw 234.32 [342789-81-5]	75-80 °C	-	100 g; 1 kg	¥25,000; ¥69,100
30764	**1-Ethyl-3-methylimidazolium chloride BASF Quality, >93%** BASIONIC™ ST 80 $C_6H_{11}ClN_2$ Mw 146.62 [65039-09-0]	80 °C	-	100 g; 1 kg	¥25,000; ¥69,100
50365	**1,2,3-Trimethylimidazolium methylsulfate BASF Quality, >95%** BASIONIC™ ST 99 $C_7H_{14}N_2O_4S$ Mw 222.26 [65086-12-6]	113 °C	-	100 g; 1 kg	¥20,000; ¥55,300
55292	**1-Butyl-3-methylimidazolium tetrachloroaluminate BASF Quality, >95%** BASIONIC™ AC 01 $C_8H_{15}AlCl_4N_2$ Mw 308.01 [80432-09-3]	-10 °C	1.2430	100 g; 1 kg	¥25,000; ¥69,100
51059	**1-Ethyl-3-methylimidazolium tetrachloroaluminate BASF Quality, >95%** BASIONIC™ AC 09 $C_6H_{11}AlCl_4N_2$ Mw 279.96 [80432-05-9]	9 °C	1.3040	100 g; 1 kg	¥30,100; ¥83,000
56486	**1-Ethyl-3-methylimidazolium hydrogensulfate BASF Quality, >95%** BASIONIC™ AC 25 $C_6H_{12}N_2O_4S$ Mw 208.24 [412009-61-1]	-	1.3673	100 g; 1 kg	¥30,100; ¥83,000
57457	**1-Butyl-3-methylimidazolium hydrogensulfate BASF Quality, >95%** BASIONIC™ AC 28 $C_8H_{16}N_2O_4S$ Mw 236.20 [262297-13-2]	28 °C	1.2770	100 g; 1 kg	¥25,000; ¥69,100
59760	**Methylimidazolium hydrogensulfate BASF Quality, >95%** BASIONIC™ AC 39 $C_4H_6N_2.H_2SO_4$ Mw 180.18 [681281-87-8]	39 °C	1.4835	100 g; 1 kg	¥22,500; ¥62,200
40477	**Methylimidazolium chloride BASF Quality, >95%** BASIONIC™ AC 75 $C_4H_6N_2.HCl$ Mw 118.56 [35487-17-3]	75 °C	-	100 g; 1 kg	¥22,500; ¥62,200
51053	**1-Ethyl-3-methylimidazolium acetate BASF Quality, >90%** BASIONIC™ BC 01 $C_8H_{14}N_2O_2$ Mw 170.22 [143314-17-4]	<-20 °C	1.0270	100 g; 1 kg	¥55,100; ¥152,100
39952	**1-Butyl-3-methylimidazolium acetate BASF Quality, >95%** BASIONIC™ BC 02 $C_{10}H_{18}N_2O_2$ Mw 198.27 [284049-75-8]	<-20 °C	1.0550	100 g; 1 kg	¥50,100; ¥138,300
51682	**1-Ethyl-3-methylimidazolium ethylsulfate BASF Quality, >95%** BASIONIC™ LQ 01 $C_8H_{16}N_2O_4S$ Mw 236.29 [342573-75-5]	<-20 °C	1.2402	100 g; 1 kg	¥20,000; ¥55,300
53177	**1-Butyl-3-methylimidazolium methylsulfate BASF Quality, >95%** BASIONIC™ LQ 02 $C_9H_{18}N_2O_4S$ Mw 250.32 [401788-98-5]	<-20 °C	1.2129	100 g; 1 kg	¥20,000; ¥55,300
43437	**1-Ethyl-3-methylimidazolium thiocyanate BASF Quality, >95%** BASIONIC™ VS 01 $C_7H_{11}N_3S$ Mw 169.25 [331717-63-6]	<-20 °C	1.1140	100 g; 1 kg	¥45,100; ¥124,400
42254	**1-Butyl-3-methylimidazolium thiocyanate BASF Quality, >95%** BASIONIC™ VS 02 $C_9H_{15}N_3S$ Mw 197.30 [344790-87-0]	<-20 °C	1.0696	100 g; 1 kg	¥40,100; ¥110,600

BASIONICS™ is a trademark of BASF AG.

イオン性液体の最新情報は、テクニカルサービス sialjpts@sial.com (日本語) へお問い合わせください。

New Ionic Liquids

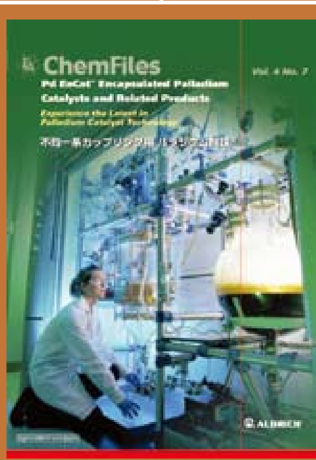
Sigma-Aldrich　別冊カタログのご紹介

NEW

イオン性液体　A-162

そのユニークな性質により近年大変注目を浴びているイオン性液体を一挙掲載。一般的なピリジニウム塩以外にホスホニウム塩、アンモニウム塩も研究が進んでいます。環境負荷の少ないハロゲンフリーのイオン性液体、Cytec社製品など、世界最大の品揃えです。

NEW



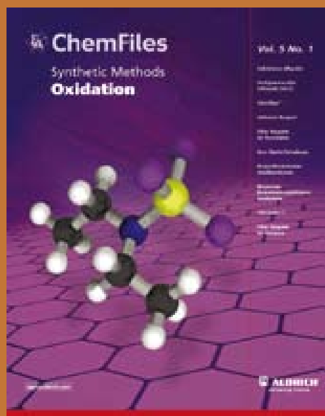
不均一系カップリング用パラジウム触媒　A-160

鈴木−宮浦反応、Heck反応、カルボニル化反応などにご利用いただけます。
主な特徴　容易な分離操作、粗生成物中の低パラジウム濃度など。

カップリング反応用試薬　A-146

Buchwaldホスフィン配位子、パラジウム触媒、Grubbs触媒、光延反応用試薬、スーパーベース、ボロン酸などカップリング反応用の必見試薬を収載。

NEW



Oxidation　A-166

石川試薬, DAST, Dess-Martin Periodinane, Iodotoluene difluorideなど種々の酸化剤、フッ素導入試薬を詳しい反応例と共に掲載した最新カタログです。

SIGMA-ALDRICH

シグマ アルドリッチ ジャパン株式会社

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