Contents lists available at ScienceDirect

Journal of Fluorine Chemistry

journal homepage: www.elsevier.com/locate/fluor

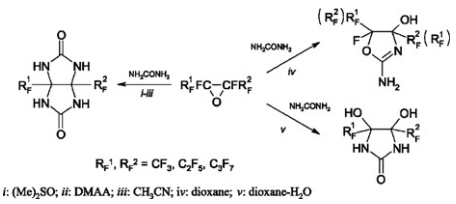
Graphical Abstracts/J. Fluorine Chem. 130 (2009) 847–852

J. Fluorine Chem., 130 (2009) 853

Synthesis of fluorine containing glycolurils and oxazolines from oxides of internal perfluoroolefins

Lyudmila V. Saloutina, Aleksandr Ya. Zapevalov, Victor I. Saloutin, Pavel A. Slepukhin, Mikhail I. Kodess, Oleg N. Chupakhin

I. Ya. Postovsky Institute of Organic Synthesis, Urals Branch of the Russian Academy of Sciences, 22/20 S. Kovalevskoy/Academicheskaya, GSP-147, 620041 Ekaterinburg, Russia



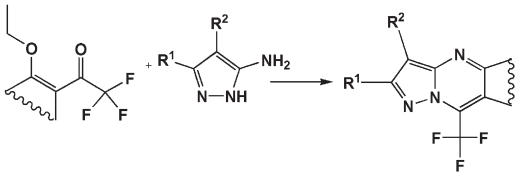
The reaction of oxides of internal perfluoroolefins with urea in polar solvents – dimethylsulfoxide, N,N-dimethylacetamide and acetonitrile – gave 1,5-bis(perfluoroalkyl) tetraazabicyclo[3.3.0]octane-3,7-diones, in moderate yields. In dioxane, unexpected cyclization occurred resulting in 2-amino-5-fluoro-4,5-bis(perfluoroalkyl)-4,5-dihydrooxazol-4-ols, in high yields. Use of aqueous dioxane resulted in mixtures of oxazolines and glycolurils as minor and 4,5-dihydroxy-4,5-bis(perfluoroalkyl)imidazilidine-2-ones as major reaction products.

J. Fluorine Chem., 130 (2009) 861

Highly regioselective synthesis of trifluoromethyl derivatives of pyrazolo[1,5-*a*]pyrimidines bearing fused cycloalkane rings using (2-ethoxycycloalkenyl)-2,2,2-trifluoroethanones

Elena E. Emelina[a], Aleksander A. Petrov[a], Stanislav I. Selivanov[a], Yulia V. Nelyubina[b], Mikhail Yu. Antipin[b]

[a]*Department of Chemistry, St. Petersburg State University, Saint-Petersburg, 198504, Russia*
[b]*A.N. Nesmeyanov Institute of Organoelemental Compounds, Russian Academy of Sciences, Moscow, 119991, Russia*



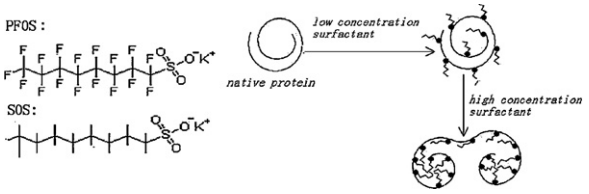
8-Trifluoromethyl-6,7-dihydro-5*H*-1,4,8a-triaza-*s*-indacene and 9-trifluoromethyl-5,6,7,8-tetrahydropyrazolo[5,1-*b*]quinazolines were efficiently generated by condensation of 5(3)-aminopyrazoles with (2-ethoxycycloalkenyl)-2,2,2-trifluoroethanones and isolated in excellent yields. The regiostructure of the prepared compounds was established by ^1H, ^{13}C and ^{19}F NMR spectroscopy and X-ray diffraction analysis.

J. Fluorine Chem., 130 (2009) 870

Bonding strength of fluorinated and hydrogenated surfactant to bovine serum albumin

Ling Li[a], Yingxi Wang[a], Gongwu Song[a], Shuilin Wu[a,b], Paul K. Chu[b], Zushun Xu[a,b]

[a]*Ministry-of-Education Key Laboratory for the Synthesis and Application of Organic Function Molecules, Hubei University, Wuhan 430062, China*
[b]*Department of Physics & Materials Science, City University of Hong Kong, Tat Chee Avenue, Kowloon, Hong Kong, China*



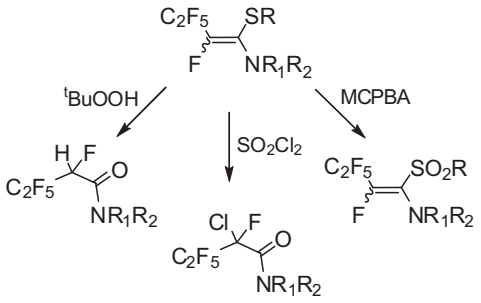
doi:10.1016/S0022-1139(09)00253-X

J. Fluorine Chem., 130 (2009) 878

Oxidation and chlorination reactions of perfluoroketene-N,S-acetals

Sergiy S. Mykhaylychenko[a,b], Jean-Philippe Bouillon[b], Yuriy G. Shermolovich[a]

[a]*Institute of Organic Chemistry, National Academy of Sciences of Ukraine, 5, Murmanska, 02094 Kiev, Ukraine*
[b]*Laboratoire Sciences et Méthodes Séparatives (SMS), EA 3233, Université de Rouen, IRCOF, F-76821 Mont-Saint-Aignan Cedex, France*



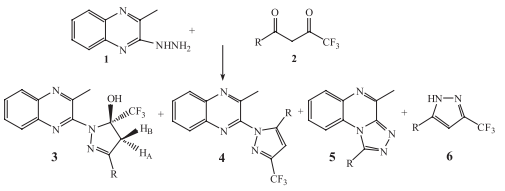
J. Fluorine Chem., 130 (2009) 886

Some novel observations on the reaction of 2-hydrazino-3-methylquinoxaline with trifluoromethyl-β-diketones

Ranjana Aggarwal, Rajiv Kumar, Shiv P. Singh

Department of Chemistry, Kurukshetra University, Kurukshetra 136 119, Haryana, India

The reaction of 2-hyrazino-3-methylquinoxaline **1** with trifluoromethyl-β-diketones **2** in different reaction conditions resulted in the formation of the unexpected products 1,2,4-triazolo[4,3-*a*]quinoxalines **5** and 3(5)-trifluoromethyl-1*H*-pyrazoles **6** in addition to the expected products 3-trifluoromethylpyrazoles, 5-hydroxy-5-trifluoromethylpyrazolines and their corresponding dehydrated pyrazoles. Also, formation of unexpected triazolo[4,3-*a*]quinoxaline **5** takes place during dehydration of hydoxypyrazolines **3** in acidic medium.



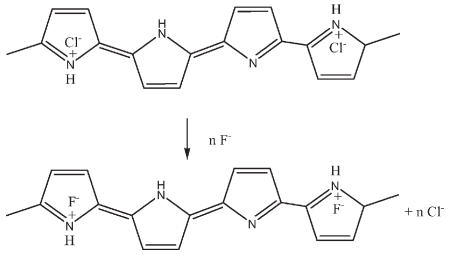
J. Fluorine Chem., 130 (2009) 894

Conducting polymer/alumina composites as viable adsorbents for the removal of fluoride ions from aqueous solution

M. Karthikeyan, K.K. Satheesh Kumar, K.P. Elango

Department of Chemistry, Gandhigram Rural University, Gandhigram 624302, India

The polyaniline/alumina (PANi–AlO) and polypyrrole/alumina (PPy–AlO) composites were prepared and characterized by FT-IR, SEM and X-ray diffraction studies and were employed as adsorbents for the removal of fluoride ions from aqueous solution by the batch sorption method.

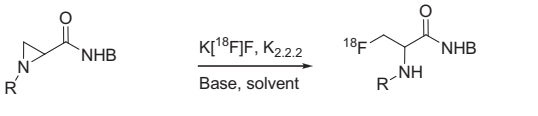


J. Fluorine Chem., 130 (2009) 902

Nucleophilic ring-opening of activated aziridines: A one-step method for labeling biomolecules with fluorine-18

Ulrike Roehn[a], Jessica Becaud[b], Linjing Mu[b], Ananth Srinivasan[a], Timo Stellfeld[a], Ansgar Fitzner[a], Keith Graham[a], Ludger Dinkelborg[a], August P. Schubiger[b], Simon M. Ametamey[b]

[a]*Bayer Schering Pharma AG, Global Drug Discovery, 13342 Berlin, Germany*
[b]*Center for Radiopharmaceutical Science of ETH, PSI and USZ, Department of Chemistry and Applied Biosciences, ETH Zurich, CH-8093, Zurich, Switzerland*



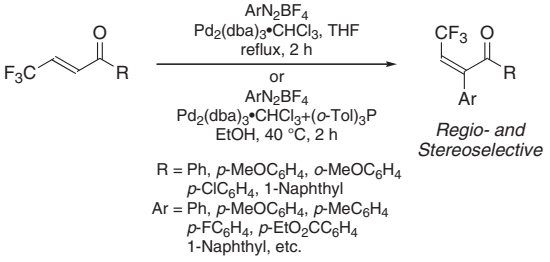
A one-step radiolabeling of model compounds and biomolecules with fluorine-18 has been achieved *via* nucleophilic ring-opening of activated aziridines (see Scheme). High to moderate yields of ^{18}F-incorporation were achieved under mild labeling conditions. One-step ^{18}F-labeling of model compounds and biomolecules via nucleophilic ring-opening of activated aziridines.

J. Fluorine Chem., 130 (2009) 913

Unexpected high regiocontrol in Heck reaction of fluorine-containing electron-deficient olefins—Highly regio- and stereoselective synthesis of β-fluoroalkyl-α-aryl-α,β-unsaturated ketones

Tsutomu Konno, Shigeyuki Yamada, Akinori Tani, Masataka Nishida, Tomotsugu Miyabe, Takashi Ishihara

Department of Chemistry and Materials Technology, Kyoto Institute of Technology, Matsugasaki, Sakyo-ku, Kyoto 606-8585, Japan



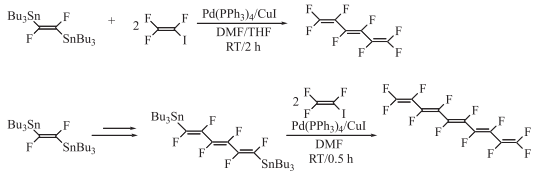
Treatment of (*E*)-4,4,4-trifluoro-1-aryl-2-buten-1-one with various aryldiazonium salts in the presence of palladium catalyst gave the corresponding α-arylated Heck adducts with high regio- and stereoselectivity in good to high yields.

J. Fluorine Chem., 130 (2009) 922

The stereoselective synthesis of (*E*)-octafluoro-1,3,5-hexatriene and (3*E*,5*E*,7*E*)-dodecafluoro-1,3,5,7,9-decapentaene

Qibo Liu, Donald J. Burton

Department of Chemistry, University of Iowa, Iowa City, IA 52242, USA



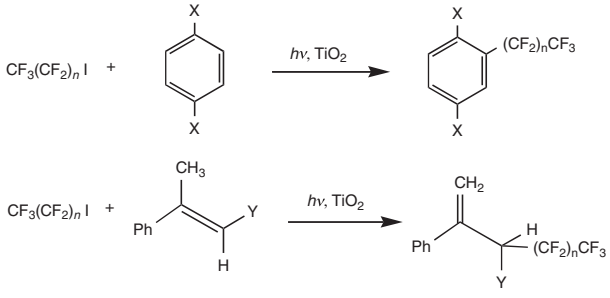
(*E*)-(1,2-Difluoro-1,2-ethenediyl)bis[tributylstannane], **3**, readily undergoes a $Pd(PPh_3)_4$/CuI catalyzed cross-coupling reaction with iodotrifluoroethene to yield (*E*)-octafluoro-1,3,5-hexatriene, 4, in high isomeric purity. (1*Z*,3*E*,5*Z*)-(1,2,3,4,5,6-Hexafluoro-1,3,5-hexenetriyl)bis[tributylstannane], **7**, was sequentially prepared from (1*Z*,3*E*,5*Z*)-(1,2,3,4,5,6-hexafluoro-1,3,5-hexenetriyl) bis[triethylsilane], **5**, which was prepared *via* a $Pd(PPh_3)_4$/CuI catalyzed cross-coupling reaction of **3** with (*E*)-1,2-difluoro-1-iodo-2-triethylsilylethene, **6**. Pd(PPh3)4/CuI cross-coupling of **7** with iodotrifluoroethene gave (3*E*,5*E*,7*E*)-dodecafluoro-1,3,5,7,9-decapentaene, **8**.

J. Fluorine Chem., 130 (2009) 926

Redox system for perfluoroalkylation of arenes and α-methylstyrene derivatives using titanium oxide as photocatalyst

Mari Iizuka, Masato Yoshida

School of Medicine, Shimane University, Izumo 693-8501, Japan

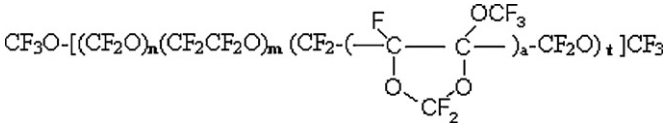


J. Fluorine Chem., 130 (2009) 933

Novel perfluoropolyethers containing 2,2,4-trifluoro-5-trifluoromethoxy-1,3-dioxole blocks: synthesis and characterization

M. Avataneo[a], W. Navarrini[b], U. De Patto[a], G. Marchionni[a]

[a]*Solvay-Solexis, R&D Centre, viale Lombardia 20, 20021 Bollate Milan, Italy*
[b]*Dipartimento di Chimica, Materiali e Ingegneria Chimica "Giulio Natta", Politecnico di Milano, via Mancinelli 7, 20131, Milan, Italy*



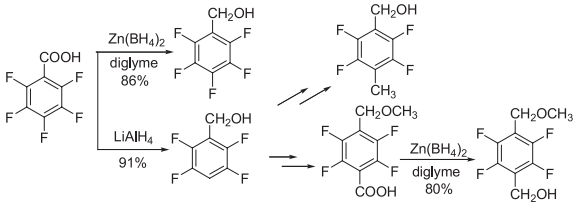
J. Fluorine Chem., 130 (2009) 938

A new synthetic route to polyfluorobenzyl alcohol

Deyan Zhang, Zizhan Chen, Huihua Cai, Xinzhuo Zou

Department of Chemistry, East China Normal University, 3663 Zhongshan Road (N), Shanghai 200062, China

The synthesis of polyfluorinated benzyl alcohol from pentafluorobenzoic acid has been developed. An economical and effective direct reduction method of polyfluorobenzoic acid by zinc borohydride is described.



J. Fluorine Chem., 130 (2009) 942

Coordination chemistry of perfluoroalkylated phosphorus(III) ligands

David Gudmunsen, Eric G. Hope, Danny R. Paige, Alison M. Stuart

Department of Chemistry, University of Leicester, Leicester LE1 7RH, UK

The spectroscopic parameters of palladium(II), platinum(II), rhodium(I), rhodium(III) and iridium(III) complexes of the tridecafluorohexyl-derivatised ligands (**L1**–**L8**), synthesized by conventional ligand displacement and/or halide-bridge cleaved reactions, are compared with those for related ligands lacking the tridecafluorohexyl ponytails.

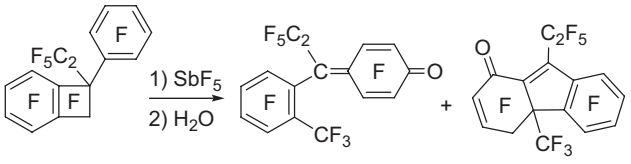
$PPh_{3-n}(C_2H_4C_6F_{13})_n$
[**L1**, n = 1; **L2,** n = 2; **L3**, n = 3]
$PPh_{3-n}(OC_2H_4C_6F_{13})_n$
[**L4**, n = 1; **L5**, n = 2; **L6**, n = 3]
$PPh_{3-n}(OC_6H_4\text{-}4\text{-}C_6F_{13})_n$
[**L7**, n = 1; **L8**, n = 2]

J. Fluorine Chem., 130 (2009) 951

Skeletal transformations of perfluoro-1-ethyl-1-phenylbenzocyclobutene in the reaction with antimony pentafluoride

Tatyana V. Mezhenkova, Victor M. Karpov, Vyacheslav E. Platonov, Yuri V. Gatilov

N.N. Vorozhtsov Novosibirsk Institute of Organic Chemistry, Novosibirsk 630090, Russia



J. Fluorine Chem., 130 (2009) 959

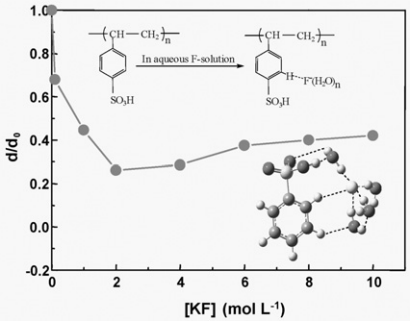
Specific F^- binding to phenyl ring of aromatic polymers

Ling Xu[a,b], Xin Li[b], Liyong Yuan[b], Maolin Zhai[b], Jing Peng[b], Jiuqiang Li[b]

[a]*Department of Energy and Resources Engineering, College of Engineering, Peking University, Beijing 100871 China*

[b]*Beijing National Laboratory for Molecular Sciences (BNLMS), Department of Applied Chemistry, College of Chemistry and Molecular Engineering, Peking University, Beijing 100871, China*

The mechanism for the abnormal reswelling of some aromatic polymer gels in concentrated KF solutions was elucidated by swelling study, elemental analysis, XPS and computational methods. The strong polarization effect of F^- and (phenyl)CH···$F^-(H_2O)_n$ interaction are two important ingredients for the reswelling of gels.

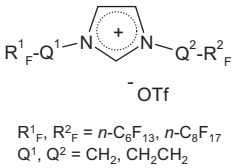


J. Fluorine Chem., 130 (2009) 966

Synthesis of bis(polyfluoroalkylated)imidazolium salts as key intermediates for fluorous NHC ligands

Martin Skalický[a], Markéta Rybáčková[a], Ondřej Kysilka[a], Magdalena Kvíčalová[b], Josef Cvačka[c], Jan Čejka[d], Jaroslav Kvíčala[a]

[a]*Department of Organic Chemistry, Institute of Chemical Technology, Prague, Technická 5, 16628 Prague 6, Czech Republic*
[b]*Institute of Inorganic Chemistry, Academy of Sciences of the Czech Republic, v.v.i., Husinec-Řež 1001, 25068 Řež, Czech Republic*
[c]*Institute of Organic Chemistry and Biochemistry, Academy of Sciences of the Czech Republic, v.v.i., Flemingovo nám. 2, 16610 Prague 6, Czech Republic*
[d]*Department of Chemistry of Solid State, Institute of Chemical Technology, Prague, Technická 5, 16628 Prague 6, Czech Republic*



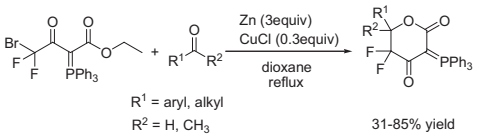
J. Fluorine Chem., 130 (2009) 974

One-pot synthesis of 4,4-difluoro-3-oxo-2-(triphenylphosphoranylidene)-δ-lactones by Reformatsky reaction

Xiang Fang[a,b], Xueyan Yang[a], Min Zhao[a], Qingfeng Di[a], Xiaoguang Wang[a], Fanhong Wu[a,b]

[a]*Laboratory for Advanced Material and Institute of Fine Chemicals, School of Chemistry and Molecular Engineering, East China University of Science and Technology, 130 Meilong Road, Shanghai 200237, China*
[b]*State Key Laboratory of Elemento-organic Chemistry, Nankai University, Tianjin 300071, China*

In the presence of zinc and a catalytic amount of cuprous chloride, ethyl 4-bromo-4,4-difluoro-3-oxo-2-(triphenylphosphoranylidene)butanoate reacted with carbonyl compounds to give the *gem*-difluoromethylenated 2-(triphenylphosphoranylidene)-δ-lactones in one-pot in moderate to high yields.



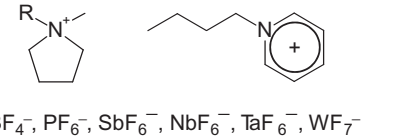
J. Fluorine Chem., 130 (2009) 979

Thermal properties of *N*-alkyl-*N*-methylpyrrolidinium and *N*-butylpyridinium fluorometallates and physicochemical properties of their melts

Takatsugu Kanatani, Ryuichi Ueno, Kazuhiko Matsumoto, Toshiyuki Nohira, Rika Hagiwara

Department of Fundamental Energy Science, Graduate School of Energy Science, Kyoto University, Sakyo-ku, Kyoto 606-8501, Japan

A series of *N*-alkyl-*N*-methylpyrrolidinium ($RMPyr^+$, where R = E: ethyl, B: butyl, and H: hexyl) and *N*-butylpyridinium (BPy^+) salts based on the fluorocomplex anions, BF_4^-, PF_6^-, SbF_6^-, NbF_6^-, TaF_6^-, and WF_7^-, have been synthesized and their thermal behavior has been investigated.

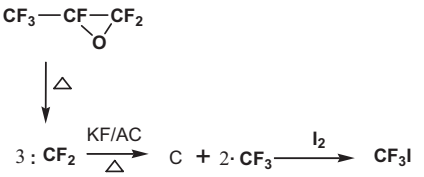


J. Fluorine Chem., 130 (2009) 985

Preparation of trifluoroiodomethane via vapor-phase catalytic reaction between hexafluoropropylene oxide and iodine

Guang-Cheng Yang[a], Xiao-Qing Jia[a], Ren-Ming Pan[a], Heng-Dao Quan[b]

[a]*School of Chemical Engineering, Nanjing University of Science and Technology, Nanjing 210094, China*
[b]*National Institute of Advanced Industrial Science and Technology (AIST), Tsukuba, Ibaraki 305-8565, Japan*

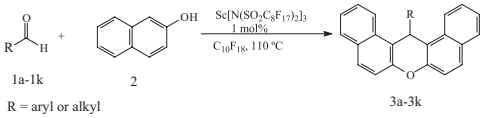


J. Fluorine Chem., 130 (2009) 989

$Sc[N(SO_2C_8F_{17})_2]_3$ catalyzed condensation of β-naphthol and aldehydes in fluorous solvent: One-pot synthesis of 14-substituted-14*H*-dibenzo[*a*,*j*]xanthenes

Mei Hong, Chun Cai

College of Chemical Engineering, Nanjing University of Science and Technology, Xiao Ling Wei Street, No. 200, Nanjing 210094, People's Republic of China



The synthesis of 14-substituted-14*H*-dibenzo[*a*,*j*]xanthenes through a condensation of β-naphthol with aldehydes was successfully accomplished using metal bis(perfluorooctanesulfonyl)imide ($M(NPf_2)n$), M = Sn, Hf, Yb, Sc, Y, Sm, Eu, Td) as catalysts in fluorous solvent. Scandium bis(perfluorooctanesulfonyl)imide catalyzed the high efficient preparation of aryl or alkyl-14*H*-dibenzo[*a*,*j*]xanthenes in fluorous solvent. Fluorous phase containing only catalyst can be reused several times.