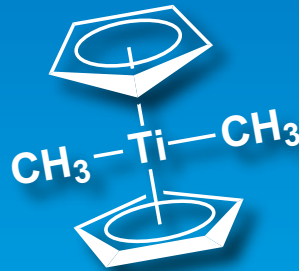
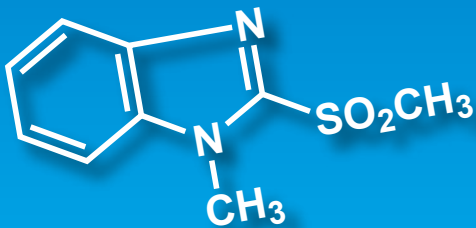
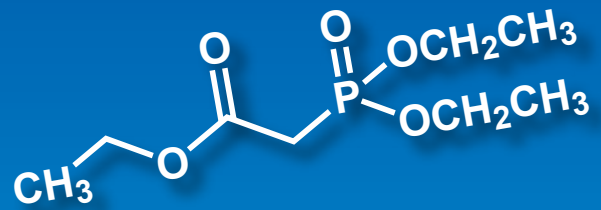
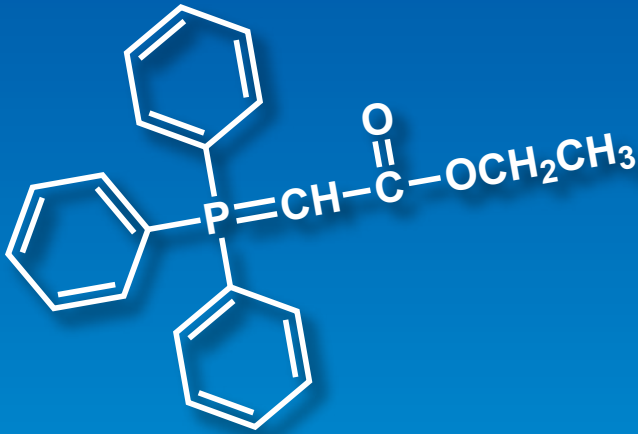


オレフィン化反応

Olefination



Wittig試薬

Horner-Wadsworth-Emmons試薬

Z選択的Horner-Wadsworth-Emmons試薬

Peterson反応試薬

Julia-Kocienskiオレフィン化試薬

チタン試薬

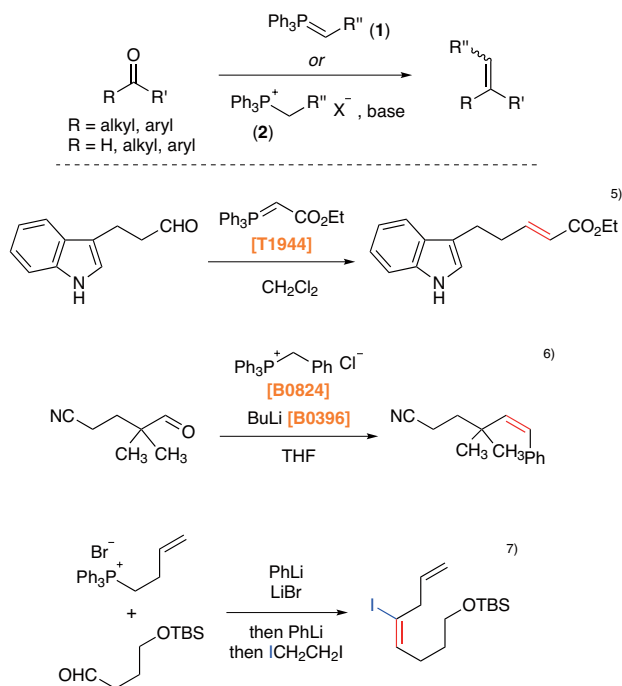
オレフィン化反応

カルボニル化合物をオレフィンに変換する方法は、有機合成上非常に重要な変換反応であり、これまでに数多くの合成手法が開発されてきました。リン原子の特性を活かしたWittig反応を筆頭に、ケイ素原子の特性を利用したPeterson反応、硫黄原子の特性を活かしたJulia-Lythgoe反応やJulia-Kocienski反応など、多くのオレフィン化反応が開発されています。特にWittig反応やHorner-Wadsworth-Emmons反応では、反応条件の改良や後処理がしやすい試薬の開発、オレフィンの二重結合の立体化学を制御する方法など、詳細な検討が進んでおり、現在も幅広く利用されています¹⁾。さらに、有機チタン化合物がエステルやアミドのカルボニル基をもメチレン化することが報告され²⁾、オレフィン化の反応の幅はより一層広がりました。

本パンフレットでは、弊社で取り扱っているオレフィン化反応に用いられるビルディングブロックを中心に記載しています。有機合成研究の一助としてお役立てください。

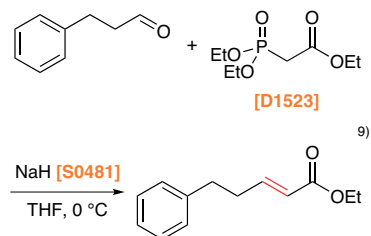
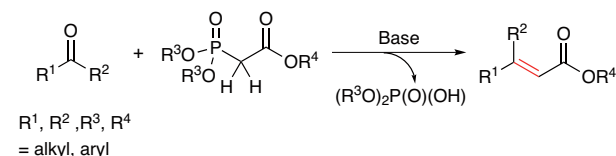
● Wittig 反応

Wittig反応は、アルデヒドやケトンにリンイリドを作用させてオレフィンを得る反応です³⁾。リンイリドには安定イリド(1)と不安定イリドがあり、不安定イリドは主にホスホニウム塩(2)に塩基を作用させて系中で発生させます。Wittig反応では主にZ体が優先して得られますが、基質や反応条件によってE体が優先する場合もあります。中でもリンイリド調製時にフェニルリチウムを用いるSchlosser法は、不安定イリドからE体を選択的に得る方法として知られています^{4,7)}。



● Horner-Wadsworth-Emmons 反応

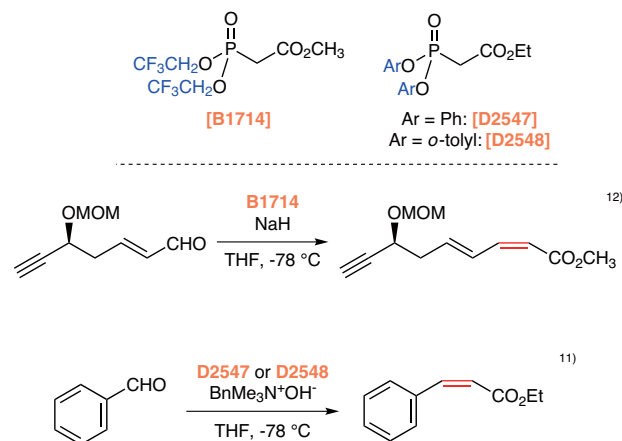
Horner-Wadsworth-Emmons (HWE)反応は、アルデヒドまたはケトンから二置換および三置換オレフィンを得る反応です⁸⁾。一般的にE体がZ体よりも優先して得られます。HWE反応では、ホスホン酸エステルと強塩基が反応して、安定かつ活性なホスホニウムアニオンが生成し、速やかにアルデヒドやケトンと反応します。副生するリン酸ジエステルは、分液操作で容易に除去可能です。



● Z 選択的

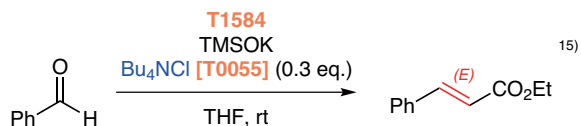
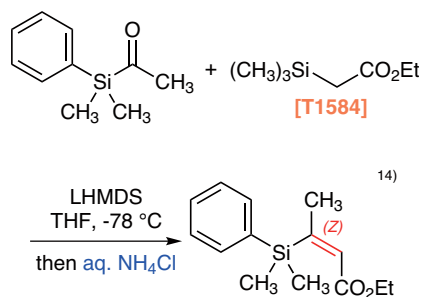
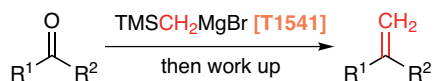
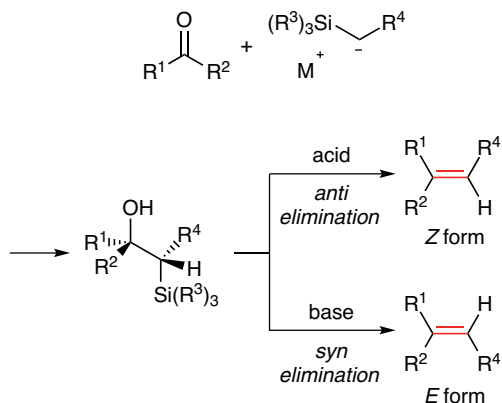
Horner-Wadsworth-Emmons 反応

HWE反応ではE体が優先して生成するため、Z体を選択的に得るには反応条件や用いるオレフィン化試薬を検討する必要があります。StillとGennariは、(ビス(ジトリフルオロエチル))ホスホノアセテート[B1714]を用いてHWE反応を行うと、Z選択的にオレフィン化が進行することを見出しました¹⁰⁾。また、安藤は、(ジアルキル)ホスホノアセテート[D2547][D2548]が高いZ選択性でα,β-不飽和エステルを与えることを見出しました¹¹⁾。特に、安藤の手法は四級アンモニウムヒドロキシドやDBUのような有機塩基も利用でき、特別な反応装置・技術を必要としないため、実用性の高いHWE試薬といえます。



● Peterson 反応

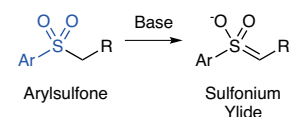
Peterson反応は、 α -シリルカルバニオンをアルデヒドやケトンに付加させ、その後に酸が塩基で処理してオレフィンを得る反応です¹³⁾。この反応は、付加体の処理の仕方によって得られるオレフィンの幾何異性が変わることが大きな特徴です。すなわち、酸処理ではシラノールが*anti*脱離してZ体が、塩基処理では*syn*脱離してE体が主生成物として得られます。



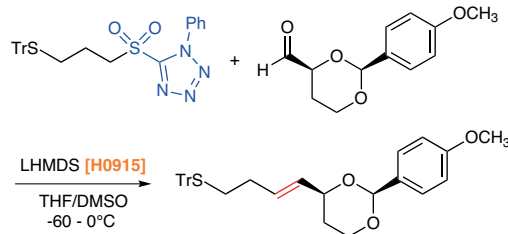
● Julia-Lythgoe 反応

Julia-Lythgoe反応は、アリールスルホンとアルデヒドがカップリングしてオレフィンを形成する反応です¹⁶⁾。この反応では、アリールスルホン由来のスルホニルイミドがアルデヒドに付加、生じた水酸基のアシル化およびNa(Hg)による還元という一連の反応でE体のオレフィンが得られます。さらに、S. A. JuliaやKocienskiらはヘテロアリールスルホンを用いた場合、ワンポットでオレフィン構築まで進行することを見出し^{17,18)}、のちに改良Julia反応と呼ばれるようになりました。この反応は、複雑な骨格をもつユニット同士のカップリングにも用いられます。また、安藤らはM2860がJulia-Kocienski型のメチレン化剤として有用であることを報告しています¹⁹⁾。

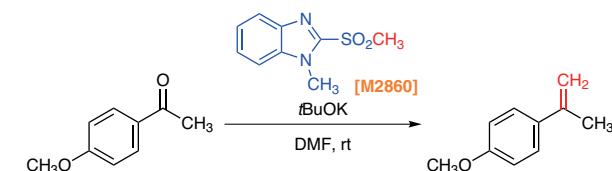
Standard Julia-Lythgoe Reaction¹⁶⁾



Julia-Kocienski Reaction²⁰⁾

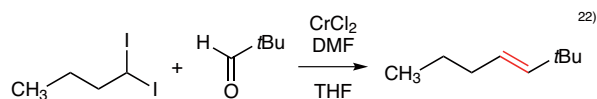
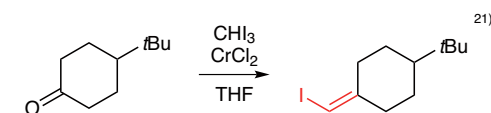
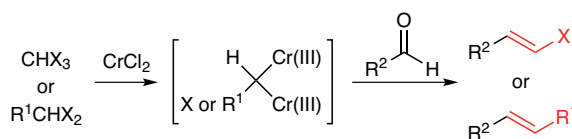


Julia-Kocienski Type Methylenylation¹⁹⁾



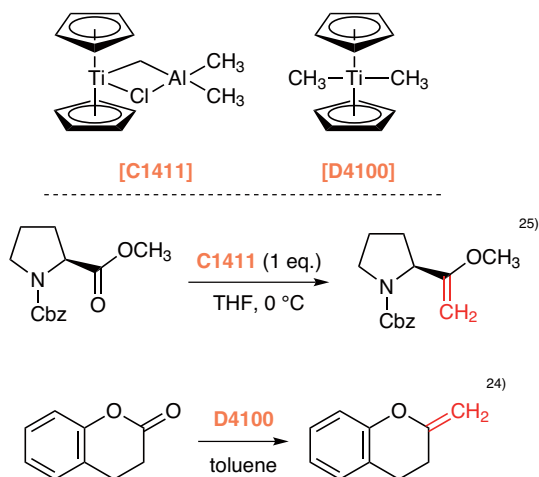
● 高井・内本反応

高井・内本らは、ハロホルムと塩化クロム(II)から調製した*gem*-ジクロム反応剤がアルデヒドと反応して、ハロオレフィンが得られることを見出しました²¹⁾。この反応は、1,1-ジハライドを用いても進行し²²⁾、アルキル鎖の伸長やほかの官能基をもつオレフィンの構築にも用いられ、主にE体のオレフィンが得られます。得られたハロオレフィンや官能基化オレフィンは、クロスカップリングなど次の反応に付すことができます。



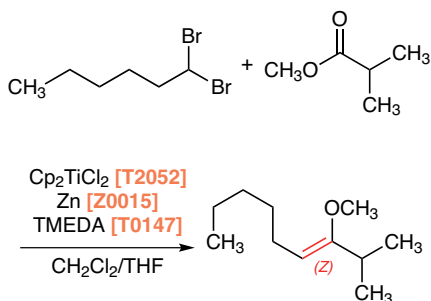
●有機チタン化合物を用いるオレフィン化反応

Tebbe試薬[C1411]²³やPetasis試薬[D4100]²⁴に代表される有機チタン化合物は、カルボニル基をエキソメチレンに変換します。両者他のオレフィン化試薬との大きな違いは、他のオレフィン化試薬はアルデヒドとケトンのみと反応するのに対し、有機チタン試薬はエステルやアミドといった比較的不活性なカルボニル基をもオレフィン化できる点です。

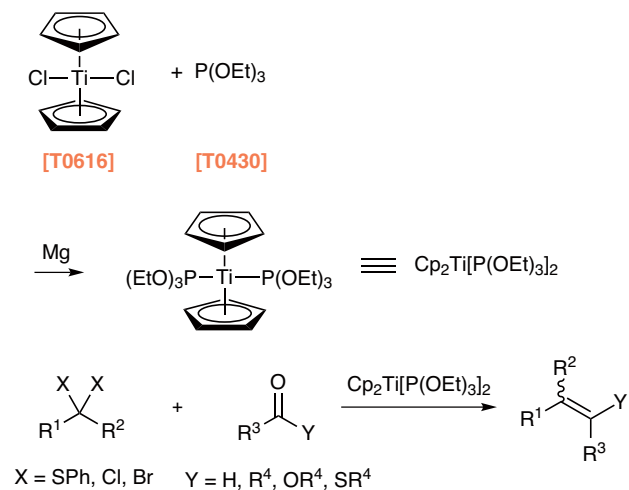


両試薬以外にも、高井・内本らはRCHX₂-TiCl₄-Zn系を用いた反応²⁶⁾を、武田らは2価チタンセンを用いるチオアセタールもしくはgem-ジハロゲン化物とアルデヒドとのオレフィン化反応²⁷⁾をそれぞれ報告しています。

Takai-Utimoto's Olefination System²⁶⁾

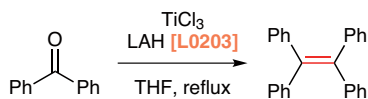


Takeda's Olefination System²⁷⁾

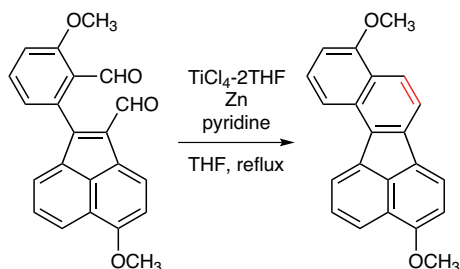


チタン試薬を用いたオレフィン化では、McMurryカップリング²⁸⁾も代表的な反応の一つといえます。この反応は、カルボニル化合物のホモカップリングだけでなく、ヘテロカップリングや分子内オレフィン化反応にも適用でき、中員環だけでなく、大員環の構築も報告されています²⁹⁾。このように、有機チタン化合物を用いるオレフィン化反応はユニークな性質と反応性をもっており、現在でも広く用いられています。

Original McMurry Coupling²⁸⁾



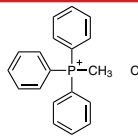
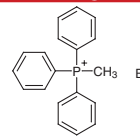
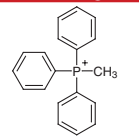
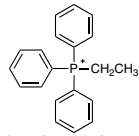
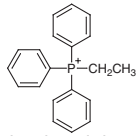
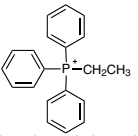
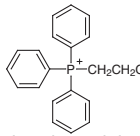
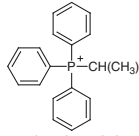
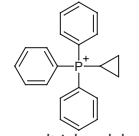
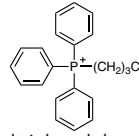
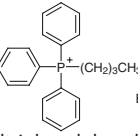
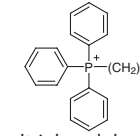
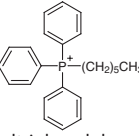
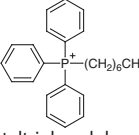
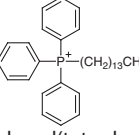
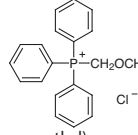
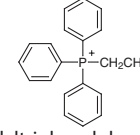
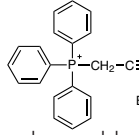
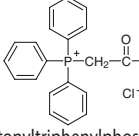
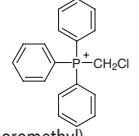
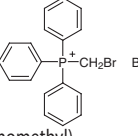
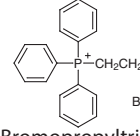
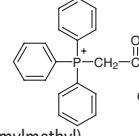
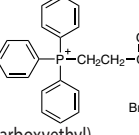
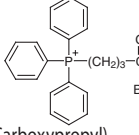
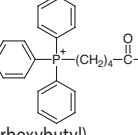
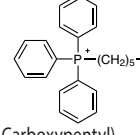
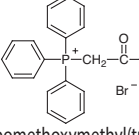
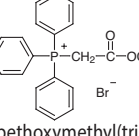
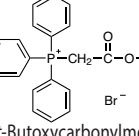
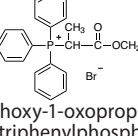
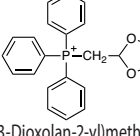
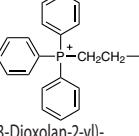
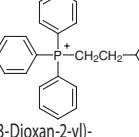
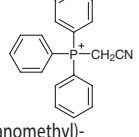
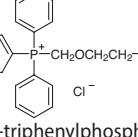
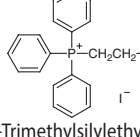
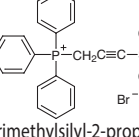
McMurry Type Ring Closure²⁹⁾

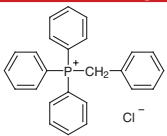
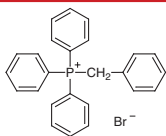
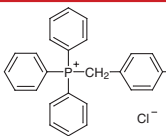
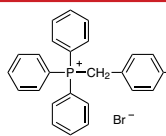
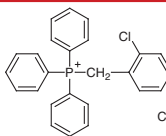
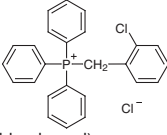
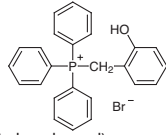
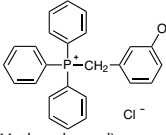
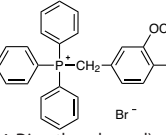
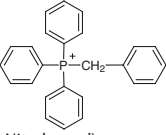
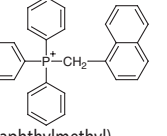
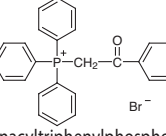
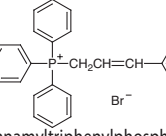
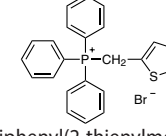
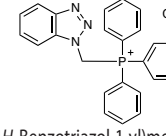
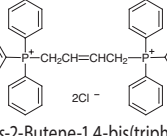
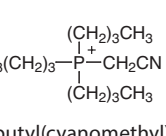
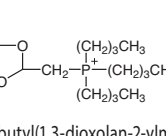
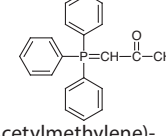
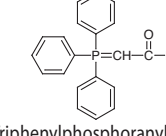
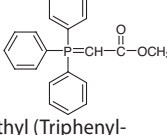
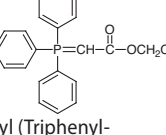
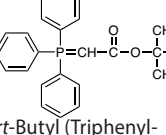
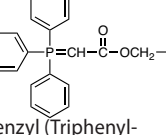
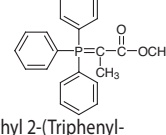
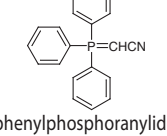
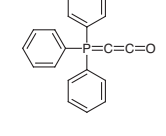
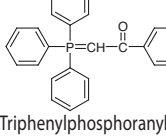
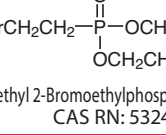
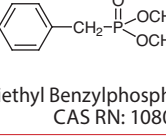
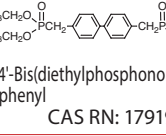
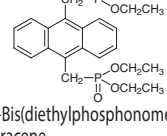
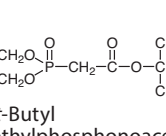
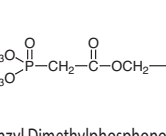


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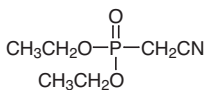
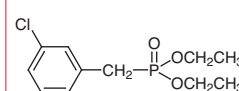
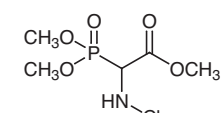
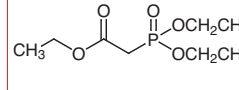
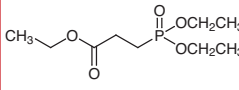
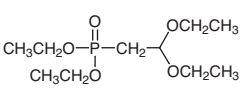
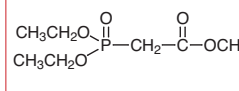
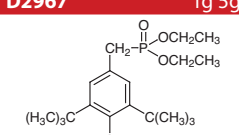
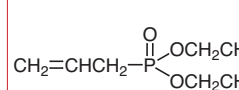
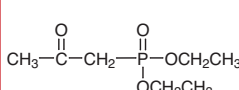
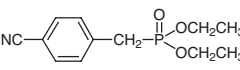
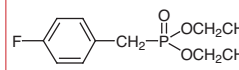
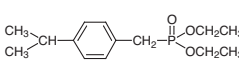
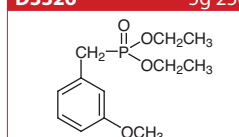
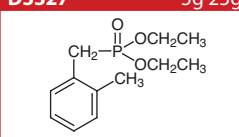
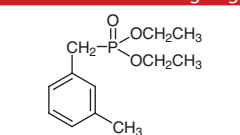
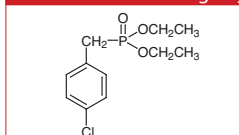
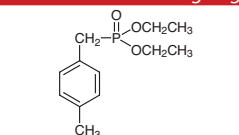
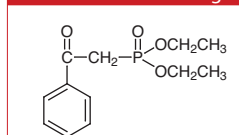
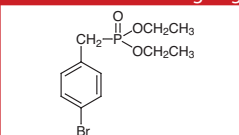
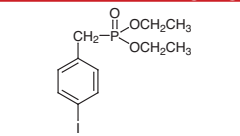
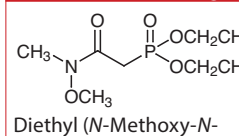
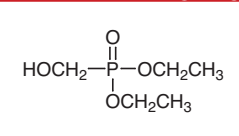
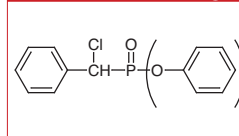
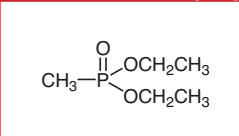
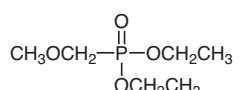
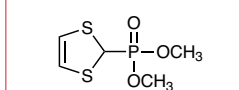
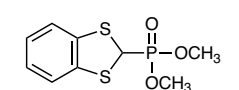
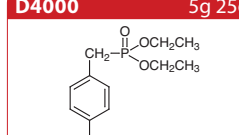
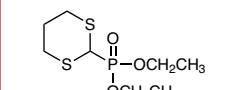
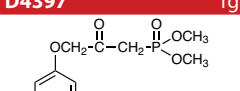
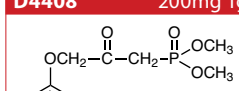



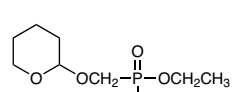
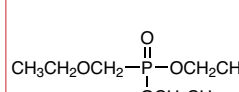
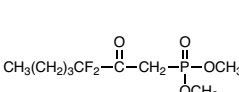
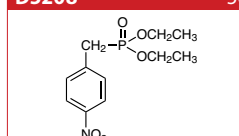
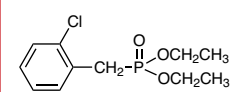
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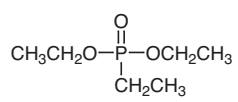
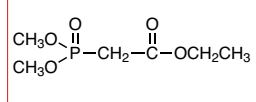
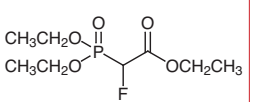
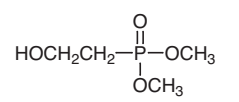
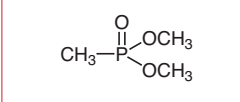
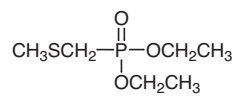
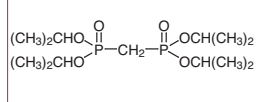
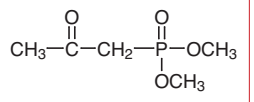
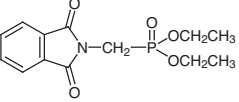
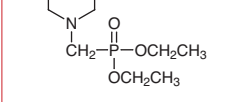
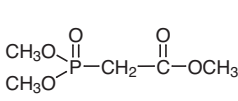
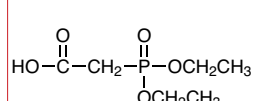
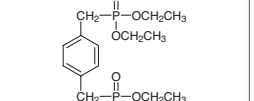
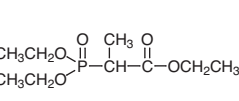
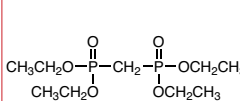
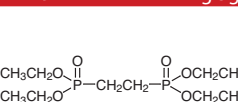
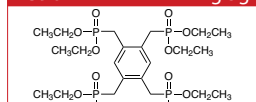
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<p>A1305 25g</p>  <p>Acetyltriphenylphosphonium Chloride CAS RN: 1235-21-8</p>	<p>C1009 5g 25g</p>  <p>(Chloromethyl)-triphenylphosphonium Chloride CAS RN: 5293-84-5</p>	<p>B1206 5g 25g</p>  <p>(Bromomethyl)-triphenylphosphonium Bromide CAS RN: 1034-49-7</p>	<p>B1208 25g</p>  <p>3-Bromopropyltriphenylphosphonium Bromide CAS RN: 3607-17-8</p>	<p>F0331 5g 25g</p>  <p>(Formylmethyl)-triphenylphosphonium Chloride CAS RN: 62942-43-2</p>
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<p>N0700 5g 25g</p>  <p>(1-Naphthylmethyl)-triphenylphosphonium Chloride CAS RN: 23277-00-1</p>	<p>P1182 25g</p>  <p>Phenacyltriphenylphosphonium Bromide CAS RN: 6048-29-9</p>	<p>C1286 25g</p>  <p>Cinnamyltriphenylphosphonium Bromide CAS RN: 7310-74-9</p>	<p>T3159 1g</p>  <p>Triphenyl(2-thienylmethyl)-phosphonium Bromide CAS RN: 23259-98-5</p>	<p>B4486 1g 5g</p>  <p>[(1H-Benzotriazol-1-yl)methyl]-triphenylphosphonium Chloride CAS RN: 111198-09-5</p>
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Horner-Wadsworth-Emmons 試薬

オレフィン化反応

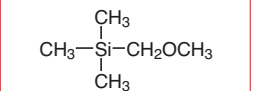
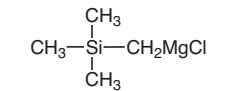
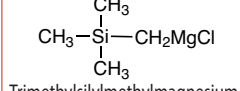
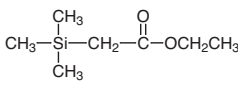
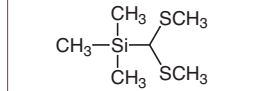
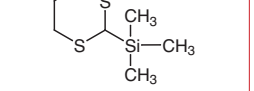
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<p>M1208 5g 25g</p>  <p>Diethyl (Methylthiomethyl)-phosphonate CAS RN: 28460-01-7</p>	<p>M1319 25g</p>  <p>Tetraisopropyl Methylenebisphosphonate CAS RN: 1660-95-3</p>	<p>O0208 5g 25g</p>  <p>Dimethyl (2-Oxopropyl)-phosphonate CAS RN: 4202-14-6</p>	<p>P1193 5g 25g</p>  <p>Diethyl (Phthalimidomethyl)-phosphonate CAS RN: 33512-26-4</p>	<p>P1258 5g</p>  <p>Diethyl 1-Pyrrolidinemethyl-phosphonate CAS RN: 51868-96-3</p>
<p>P1265 25g 250g</p>  <p>Diethyl Carboxymethylphosphonate CAS RN: 5927-18-4</p>	<p>D3080 5g 25g</p>  <p>2-(Diethoxyphosphoryl)-acetic Acid CAS RN: 3095-95-2</p>	<p>T1582 5g 25g 100g</p>  <p>Tetraethyl <i>p</i>-Xylylenediphosphonate CAS RN: 4546-04-7</p>	<p>T2135 5g 25g</p>  <p>Triethyl 2-Phosphonopropionate CAS RN: 3699-66-9</p>	<p>T2329 5g 25g</p>  <p>Tetraethyl Methylenebis(phosphonate) CAS RN: 1660-94-2</p>
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Z 選択的 Horner-Wadsworth- Emmons 試薬

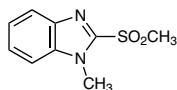
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Peterson 反応試薬

<p>M1264 5mL</p>  <p>Methoxymethyltrimethylsilane CAS RN: 14704-14-4</p>	<p>T1451 100mL</p>  <p>Trimethylsilylmethylmagnesium Chloride (20% in Ethyl Ether, ca. 1mol/L) CAS RN: 13170-43-9</p>	<p>T2609 100mL</p>  <p>Trimethylsilylmethylmagnesium Chloride (ca. 18% in Tetrahydrofuran, ca. 1mol/L) CAS RN: 13170-43-9</p>
<p>T1584 5g 25g</p>  <p>Ethyl (Trimethylsilyl)-acetate CAS RN: 4071-88-9</p>	<p>B2004 1g</p>  <p>Bis(methylthio)-(trimethylsilyl)methane CAS RN: 37891-79-5</p>	<p>T1514 5g 25g</p>  <p>2-Trimethylsilyl-1,3-dithiane CAS RN: 13411-42-2</p>

Julia-Kocienski オレフィン化試薬

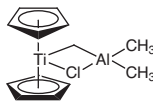
M2860 1g 5g



1-Methyl-2-(methylsulfonyl)-
benzimidazole
CAS RN: 61078-14-6

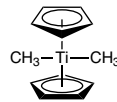
チタン試薬

C1411 25mL



Tebbe Reagent
(ca. 0.5mol/L in Toluene)
CAS RN: 67719-69-1

D4100 25g 100g



Petasis Reagent
(5% in Tetrahydrofuran/Toluene)
CAS RN: 1271-66-5

T0616 5g 25g



Titanocene Dichloride
CAS RN: 1271-19-8

T2052 100mL 500mL



Titanium(IV) Chloride (14% in
Dichloromethane, ca. 1.0mol/L)
CAS RN: 7550-45-0

T3238 100mL 500mL



Titanium(IV) Chloride
(ca. 19% in Toluene, ca. 1.0mol/L)
CAS RN: 7550-45-0

東京化成工業株式会社

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