朱传娴

客户顾问

hzhu@acs-i.org

SciFinder反应检索难点解析

中国药科大学

SCIFINDER®
A CAS SOLUTION

提纲

- SciFinder反应检索难点解析
  - 反应的精准定义
  - 巧妙获得有实验步骤的相关反应
  - 如何获得最全面的合成信息
  - 反应信息太少时如何获得更多的反应信息
  - 结果集庞大时如何限定反应
  - 直接检索反应受限时如何处理
  - 如何排除不要的反应
  - 新化合物的合成路线设计
  - 案例分析
  - 反应检索上机练习

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SciFinder检索选项——反应检索

- 反应检索方法

  结构式

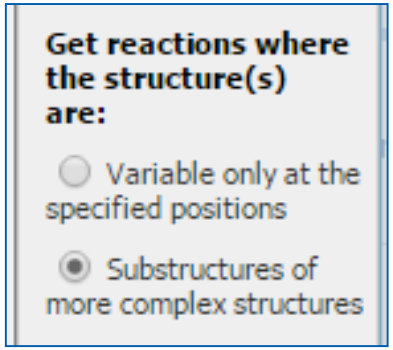
- 常用获取方法

  已知物质：由物质获取反应

  已知文献：从文献中获取反应

  精确结构反应检索

  亚结构反应检索



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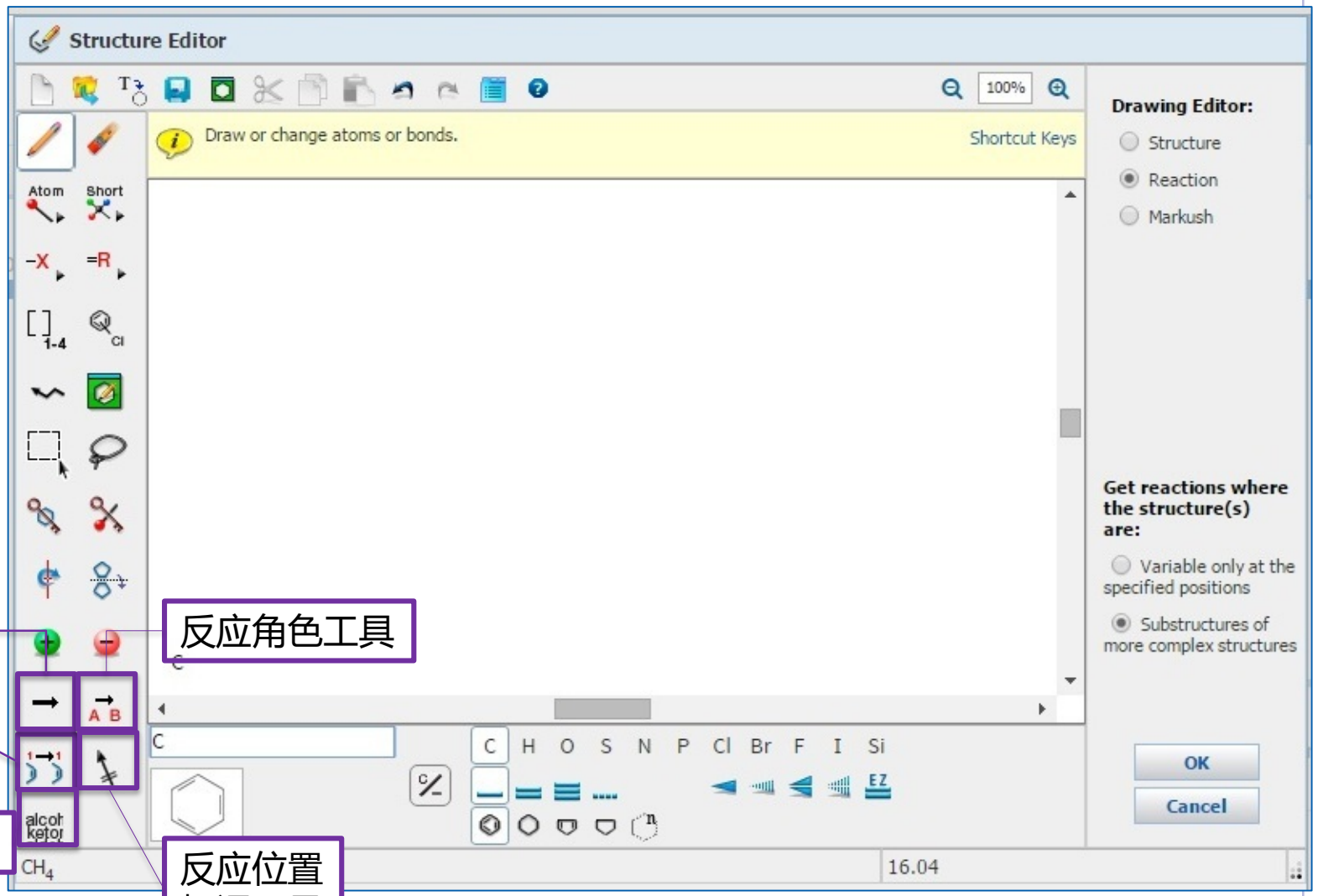
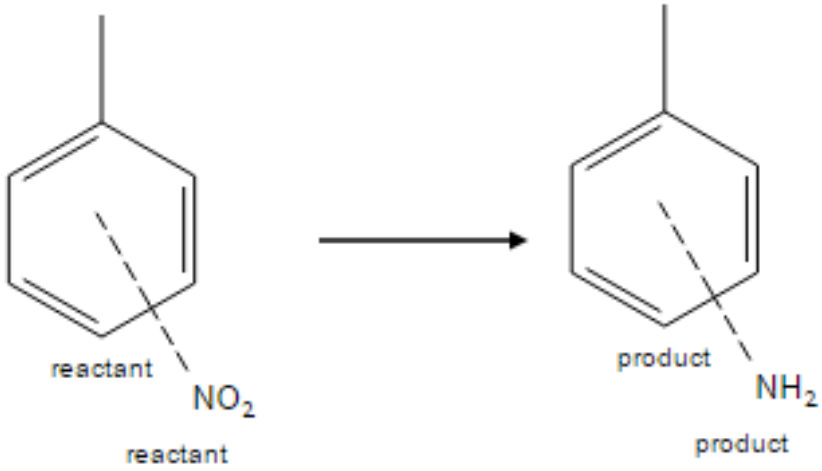
反应绘制工具


Structure Editor
100%
Draw or change atoms or bonds.
Shortcut Keys
Drawing Editor:
Structure
Reaction
Markush
Get reactions where the structure(s) are:
Variable only at the specified positions
Substructures of more complex structures
OK
Cancel
C H O S N P Cl Br F I Si
CH4
16.04
反应箭头
反应角色工具
反应原子标记工具
官能团列表
反应位置标记工具

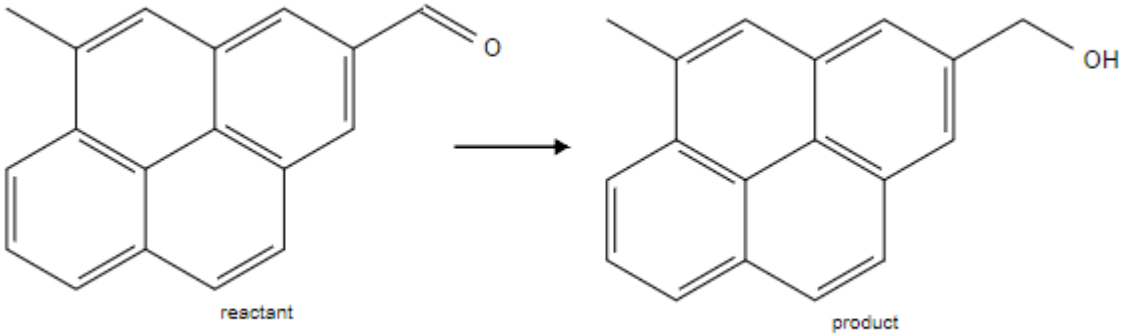
反应的精准定义

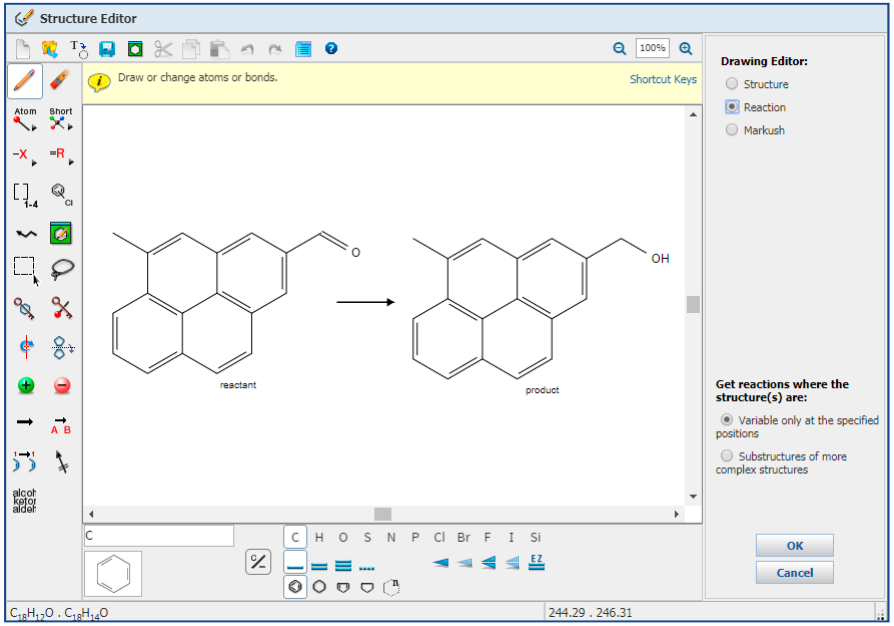
检索符合以下要求的反应，硝基还原

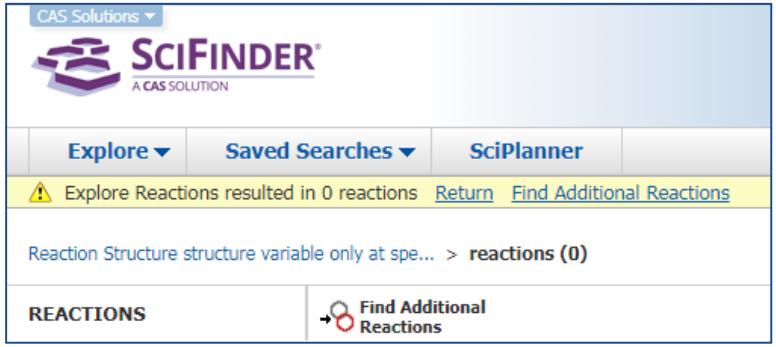


巧妙获得有实验步骤的相关反应

该还原反应如何操作？







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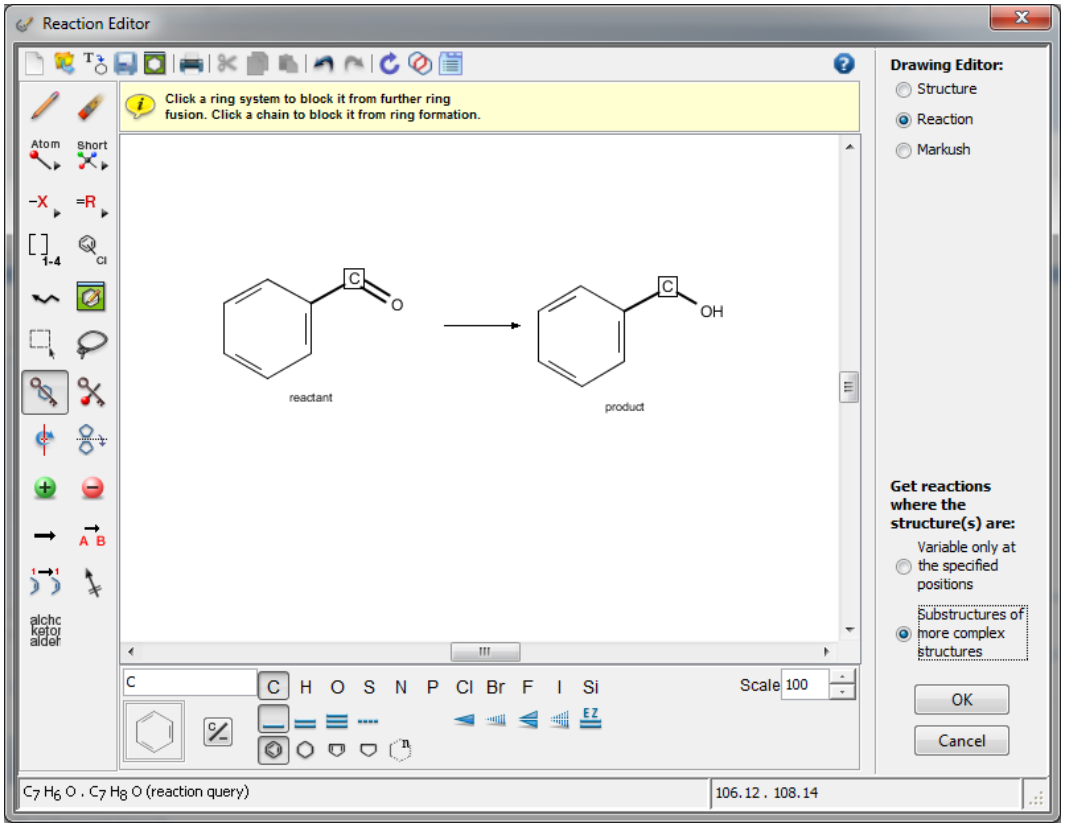
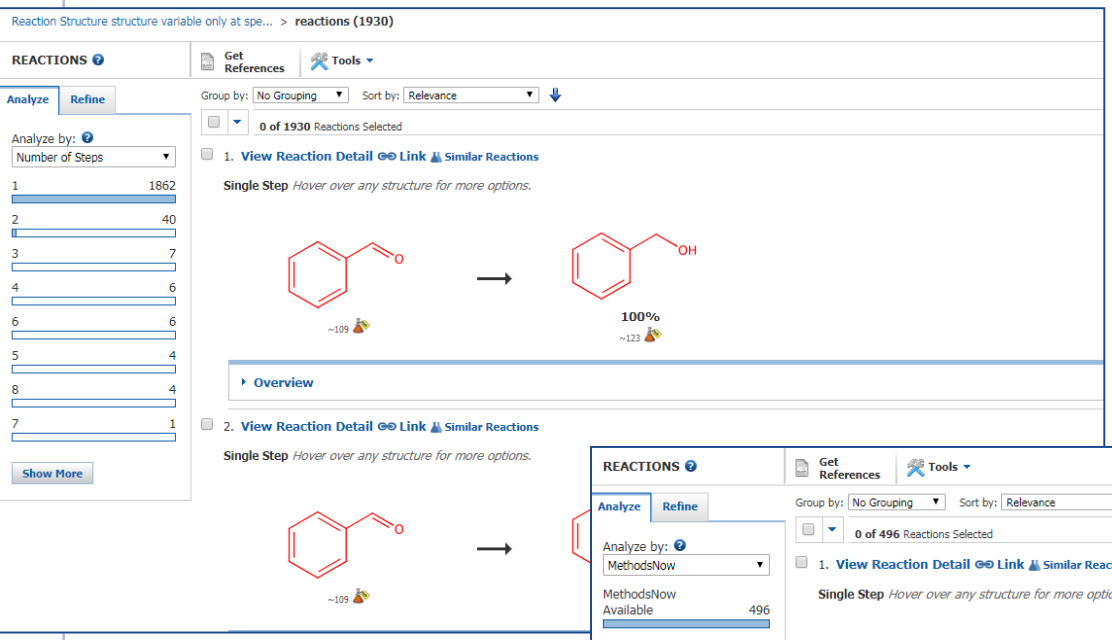
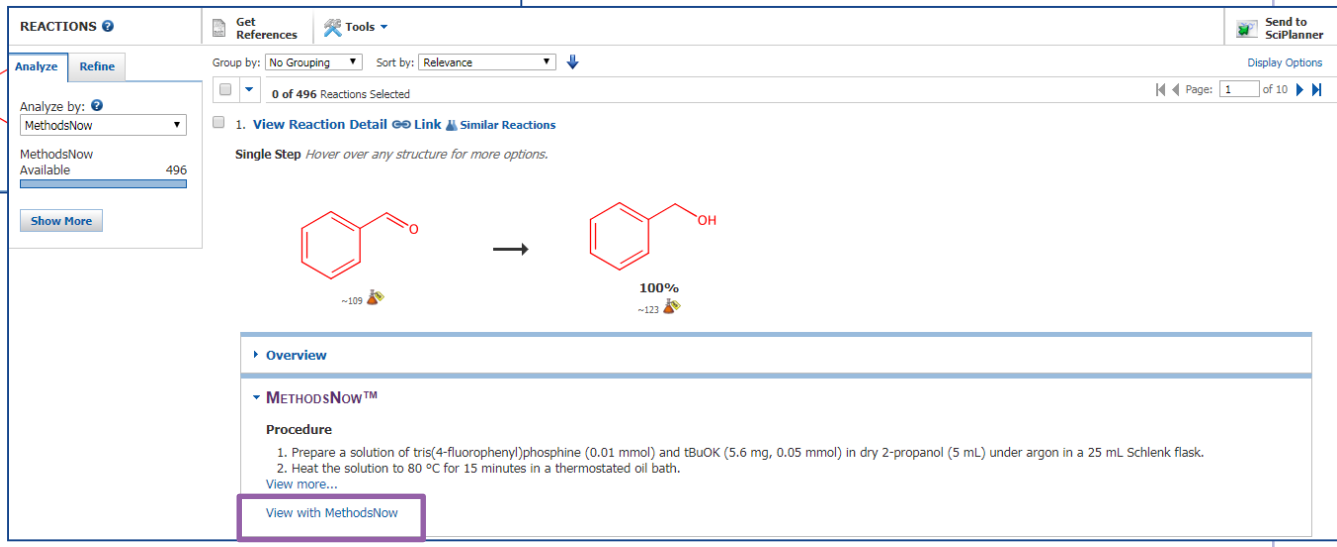
巧妙获得有反应步骤的相关反应



聚焦反应关键部位，查找类似反应

巧妙获得有反应步骤的相关反应





通过Refine获得单步反应后选取有实验步骤的反应

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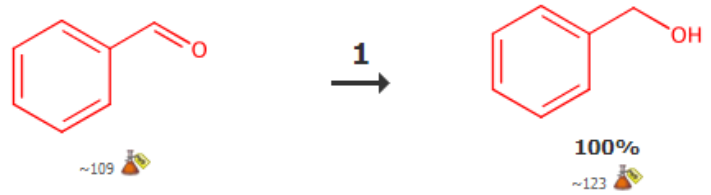
巧妙获得有反应步骤的相关反应

MethodsNow

N-Heterocyclic olefins as ancillary ligands in catalysis: a study of their behaviour in transfer hydrogenation reactions

By Iturmendi, Amaia; Garcia, Nestor; Jaseer, E. A.; Munarriz, Julen; Sanz Miguel, Pablo J.; Polo, Victor; Iglesias, Manuel; Oro, Luis A.
From Dalton Transactions, 45(32), 12835-12845; 2016
Published by Royal Society of Chemistry



Products	Benzyl alcohol, 100%, CAS RN: 100-51-6
Reactants	Benzaldehyde, CAS RN: 100-52-7
Reagents	Isopropanol, CAS RN: 67-63-0 Potassium *tert*-butoxide, CAS RN: 865-47-4
Catalysts	CAS RN: 1801329-25-8 Tris(4-fluorophenyl)phosphine, CAS RN: 18437-78-0
Solvents	Tetrahydrofuran, CAS RN: 109-99-9
Procedure	1. Prepare a solution of tris(4-fluorophenyl)phosphine (0.01 mmol) and tBuOK (5.6 mg, 0.05 mmol) in dry 2-propanol (5 mL) under argon in a 25 mL Schlenk flask. 2. Heat the solution to 80 °C for 15 minutes in a thermostated oil bath. 3. Add benzaldehyde (1 mmol) and mesitylene (140 µL, 1 mmol) to the mixture. 4. Stir the resulting mixture at 80 °C. 5. Monitor the reaction by gas chromatography.

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获得全面的合成信息

查找合成Daclatasvir的反应

获得全面的合成信息

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Substance Identifier "Daclatasvir " > substances (1) > get reactions (50) > get references (11)

REFERENCES | Get Substances | Get Reactions | Get Related Citations | Tools | This functionality is not available for this answer set. | Create Keep Me Posted Alert | Send to SciPlanner

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Sort by: Accession Number | Display Options

Analyze by: Author Name

Feng Zhiyong 2
Zhou Shengfeng 2
Bai Shunqiang 1
Cai Jin 1
Chava Satyanarayana 1
Chen Bo 1
Chen Hui 1
Chen Junqing 1
Chen Long 1

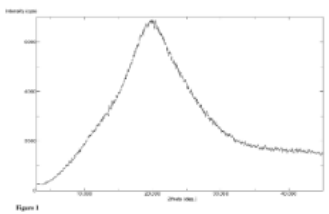
0 of 11 References Selected

1. Process for the preparation of daclatasvir, its oxalate and dihydrochloride salts

Quick View | PATENTPAK

By Swargam, Sathyanarayana; Medikonduri, Sreekanth; Salla, Rammohan; Indukuri, Venkata Sunil Kumar; Kalidindi, Srihari Raju; Chava, Satyanarayana

From PCT Int. Appl. (2017), WO 2017021904 A1 20170209. | Language: English, Database: CAPLUS



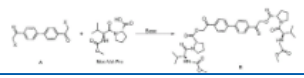
The invention is related to a process for the prepn. of daclatasvir (I) and its pharmaceutically acceptable salts by: (a) reacting biphenyl with $XCOCH_2X$ in the presence of a suitable Lewis acid; (b) reacting $XCH_2CO\text{-}p\text{-}C_6H_4\text{-}p\text{-}C_6H_4\text{-}COCH_2X$ (II) [X = halogen] with III [PG = protecting group] in the presence of a base in an org. solvent; (c) cyclization of IV; (d) deprotecting the PG of IV; converting V to I or one of its pharmaceutically acceptable salts. The invention is also relates to a process for the prepn. and purifn. of daclatasvir dihydrochloride. Thus, acylation of biphenyl with chloroac...

2. A synthetic method of daclatasvir

Quick View | PATENTPAK

By Yan, Qing; Xie, Hai; Chen, Wenrui; Bai, Shunqiang; Zhang, Li

From Faming Zhuanli Shenqing (2016), CN 106256825 A 20161228. | Language: Chinese, Database: CAPLUS



The present invention provides a synthetic method of daclatasvir, which comprises: taking 4,4'-bis(2-halogenated acetyl)biphenyl as raw material, carrying out esterification reaction with N-(methoxycarbonyl)-L-valine-L-proline in the presence of org. solvent and alkali to obtain intermediate B; then, carrying out cyclodehydration reaction on intermediate B with ammonium acetate to obtain

还有其他文献描述了目标化合物的合成吗？

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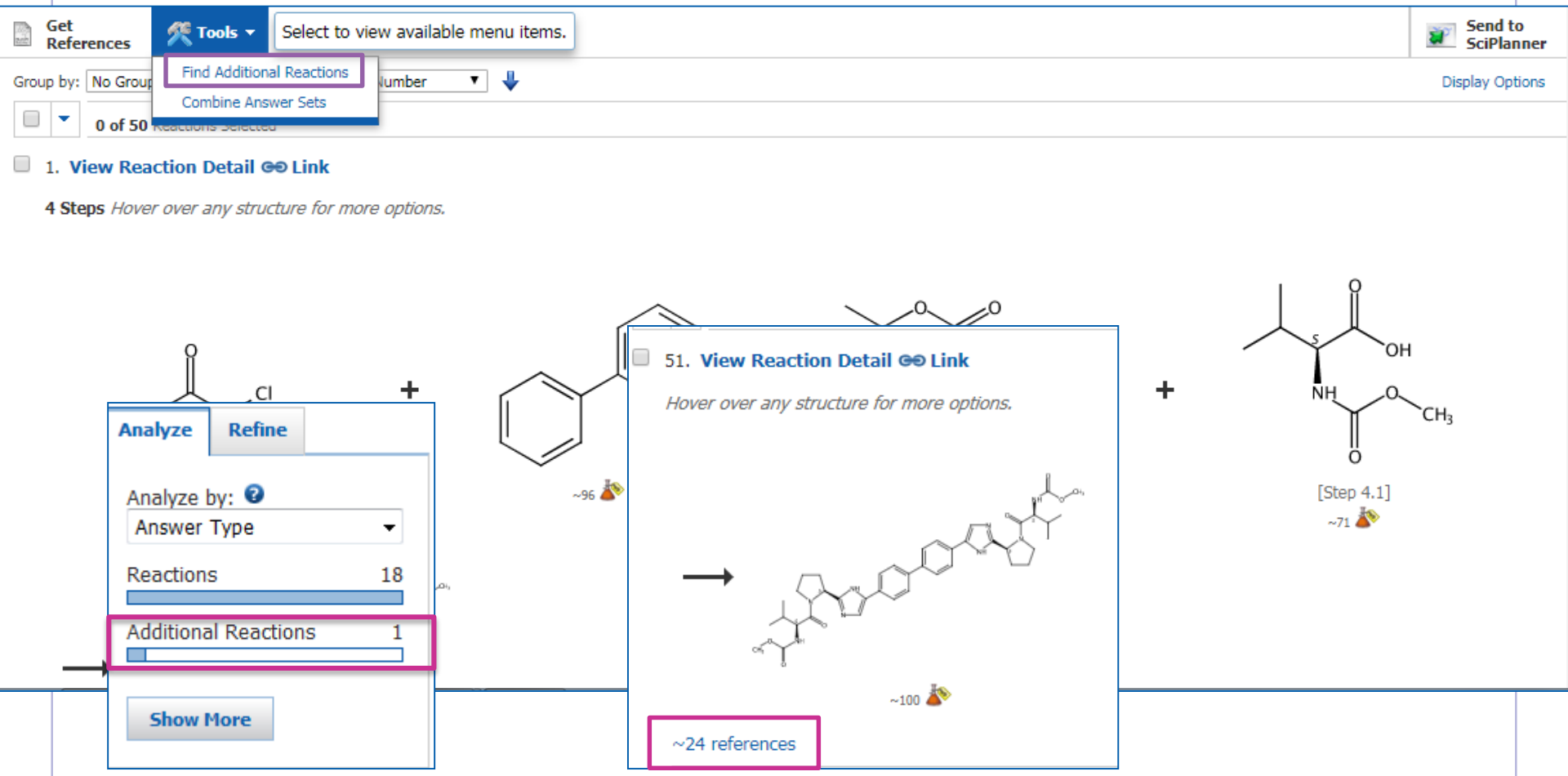
获得全面的合成信息



获得的附加反应不是完整反应

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Substance Identifier "Daclatasvir " > substances (1) > get reactions (50) > get references (11) > **Combine Reference Answer Sets "24 (24)" (32)**

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- Yang Fukang 6
- Belema Makonen 5
- Nguyen Van N 5
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- Snyder Lawrence B 4
- St Laurent Denis R 4
- Bachand Carol 3
- Good Andrew C 3
- Hamann Lawrence

Sort by: Accession Number | Display Options

0 of 32 References Selected | Page: 1 of 2

1. **A process for preparing daclatasvir and its intermediates**

Quick View | PATENTPAK

By Ying, Shuhuan; Pi, Hongjun; Wang, Liang; Chen, Jian

From Faming Zhuanli Shenqing (2017), CN 106496199 A 20170315. | Language: Chinese, Database: CAPLUS

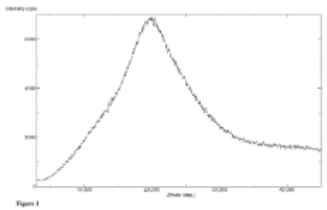
The invention relates to a process for the prepn. of daclatasvir and its intermediates. For instance, daclatasvir was prepd. via substitution of 4,4'-bis(2-bromoacetyl)biphenyl with N-Boc-L-proline in the presence of DIPEA/CH_3CN followed by intramol. heterocyclization in the presence of NH_4OAc, deprotection, and amidation with N-(methoxycarbonyl)-L-valine.

2. **Process for the preparation of daclatasvir, its oxalate and dihydrochloride salts**

Quick View | PATENTPAK

By Swargam, Sathyanarayana; Medikonduri, Sreekanth; Salla, Rammohan; Indukuri, Venkata Sunil Kumar; Kalidindi, Srihari Raju; Chava, Satyanarayana

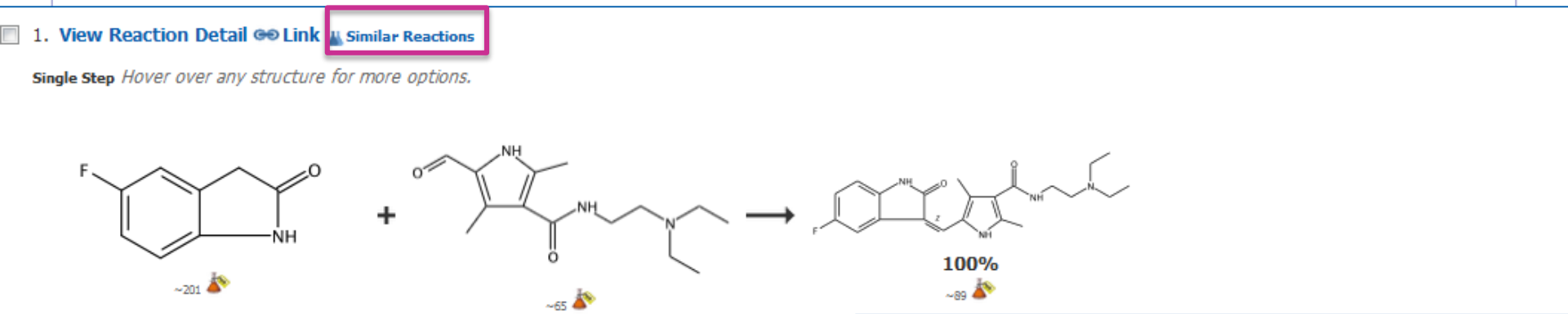
From PCT Int. Appl. (2017), WO 2017021904 A1 20170209. | Language: English, Database: CAPLUS

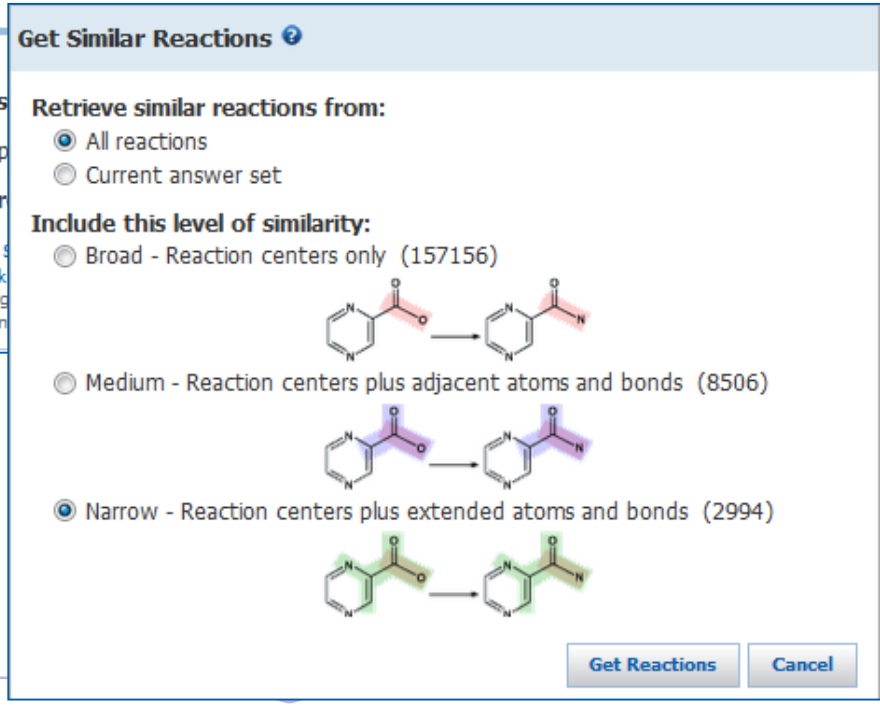


The invention is related to a process for the prepn. of daclatasvir (I) and its pharmaceutically acceptable salts by: (a) reacting biphenyl with $XCOCH_2X$ in the presence of a suitable Lewis acid; (b) reacting XCH_2CO-p-C_6H_4-p-C_6H_4-$COCH_2X$ (II) [X = halogen] with III [PG = protecting group] in the presence of a base in an org. solvent; (c) cyclization of IV; (d) deprotecting the PG of IV; converting V to I or one of its pharmaceutically acceptable salts. The invention is also relates to a process for the prepn. and purifn. of daclatasvir dihydrochloride. Thus, acylation of biphenyl with chloroac...

合并后的文献结果集

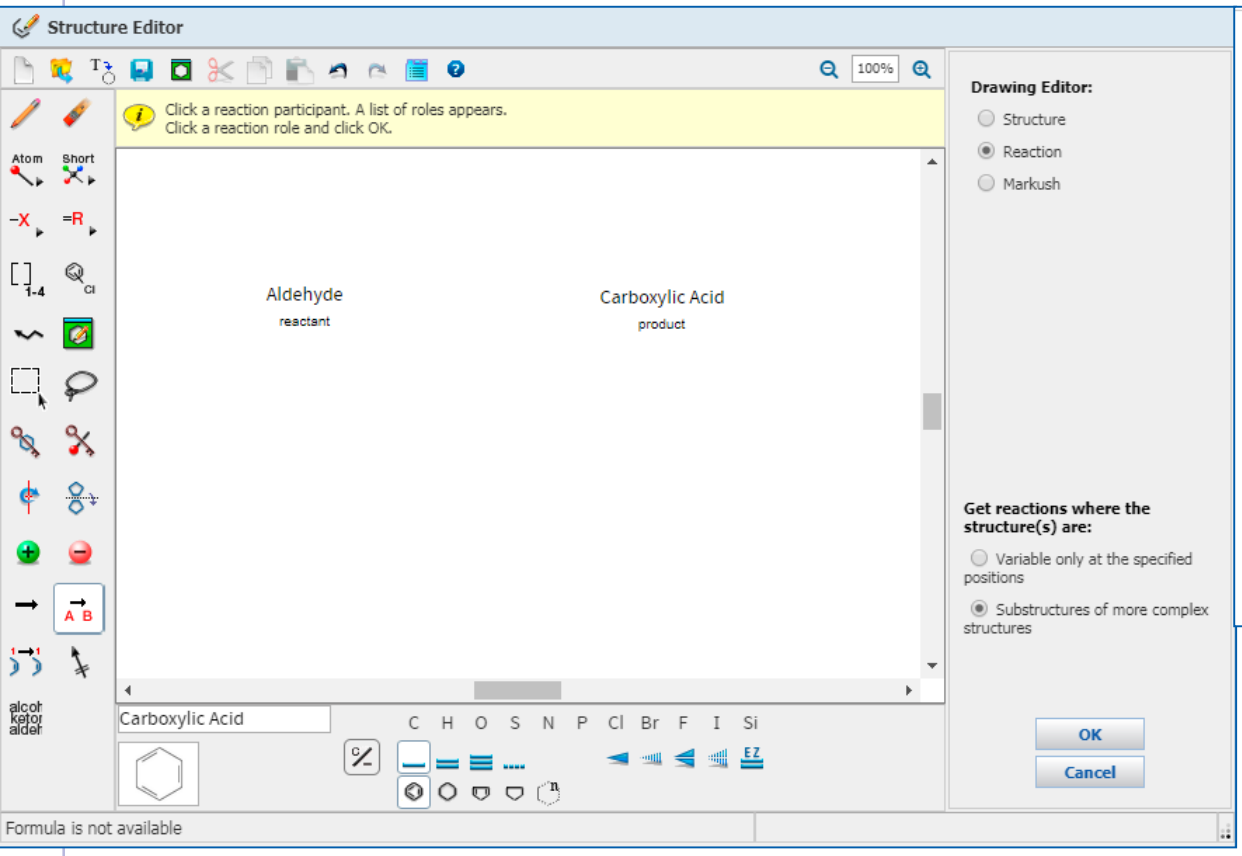
获得相关的反应信息





反应信息太少，通过Similar Reaction
获得更多反应信息

反应结果集庞大时如何限定—反应试剂的限定



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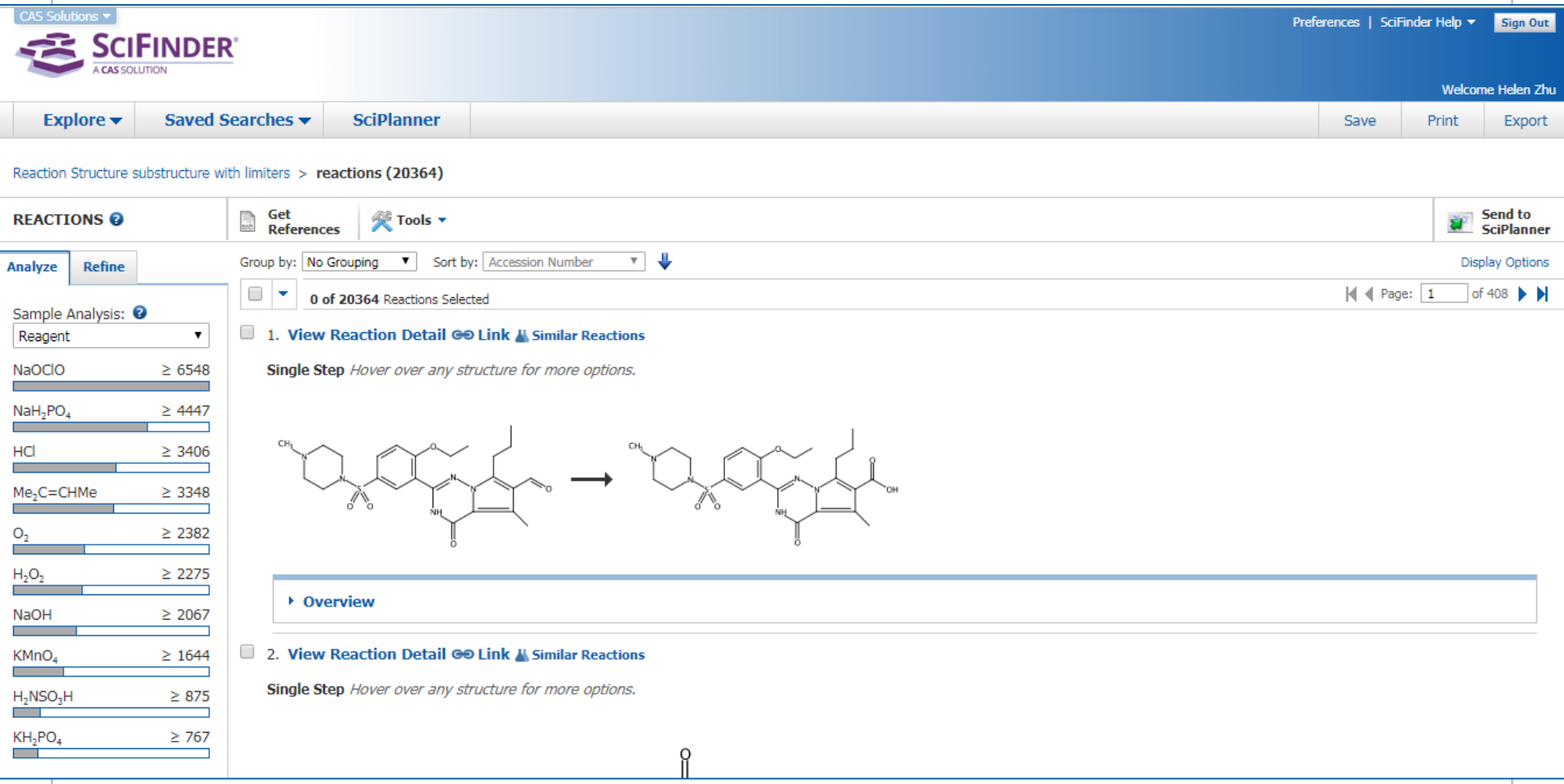
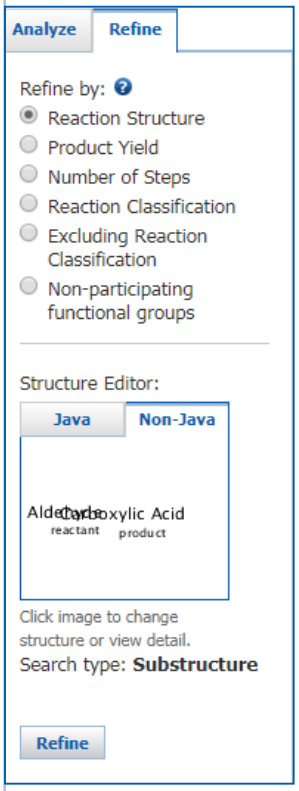
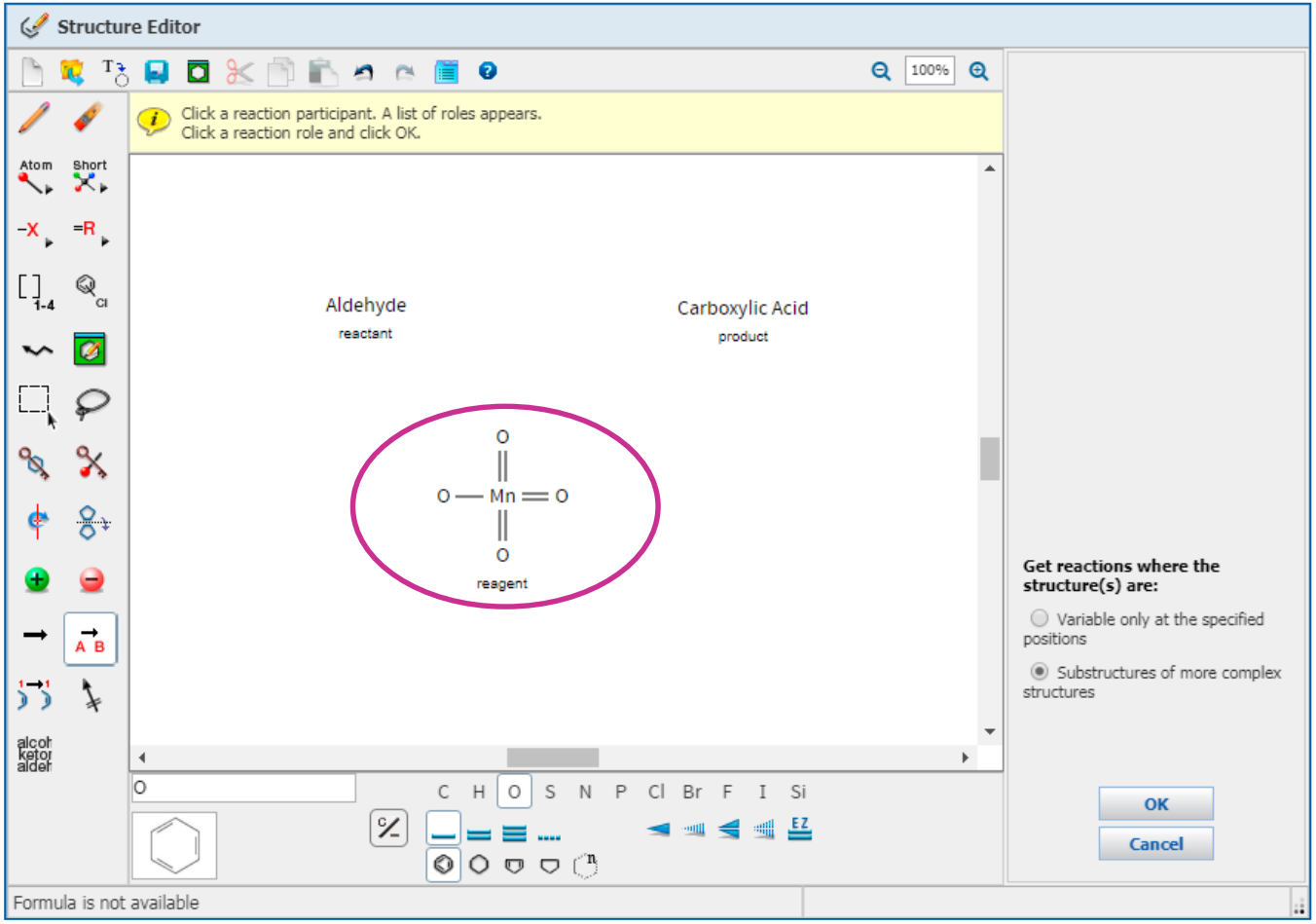
反应结果集庞大时如何限定—反应试剂的限定

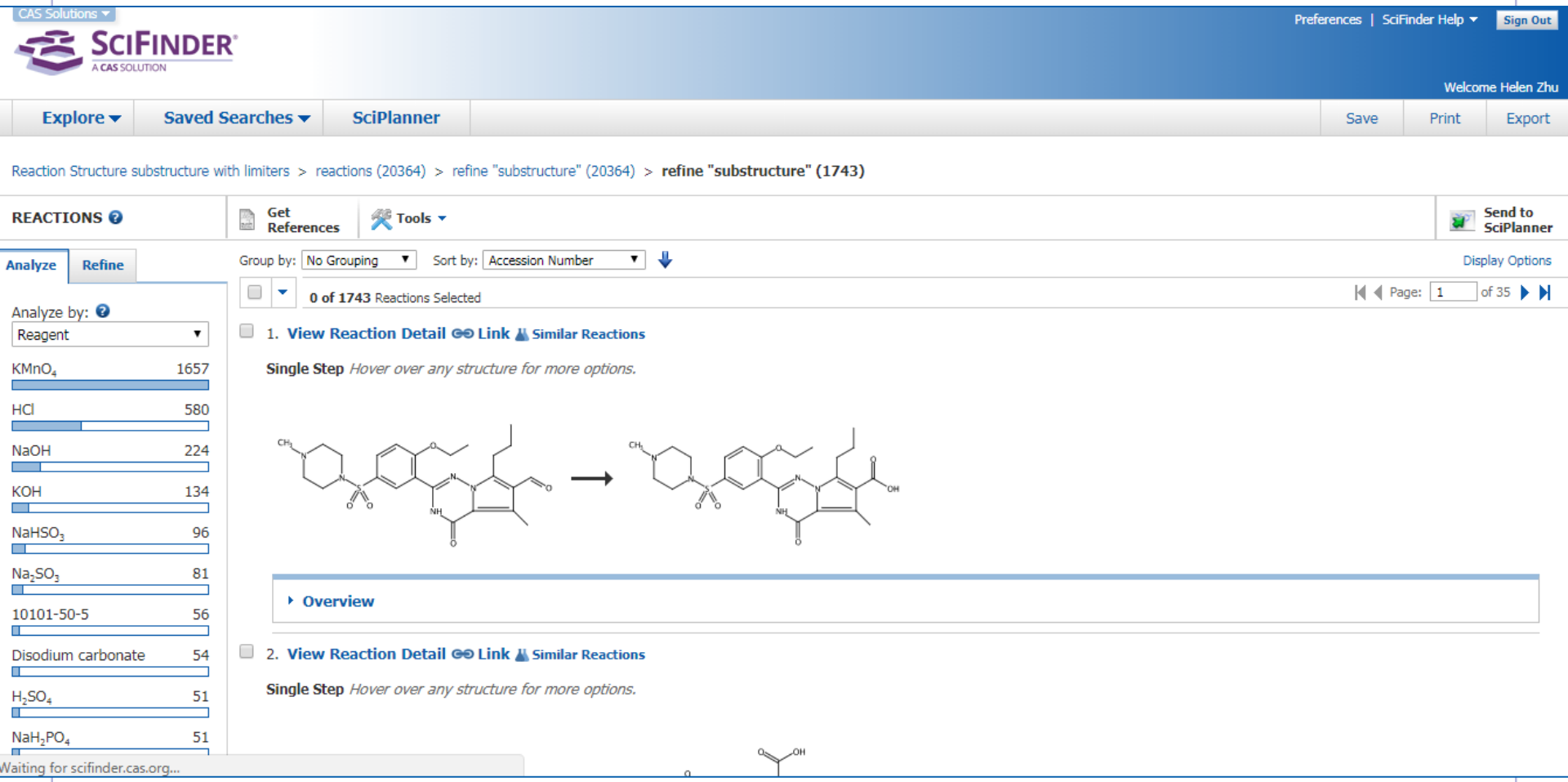


反应结果集庞大时如何限定—反应试剂的限定





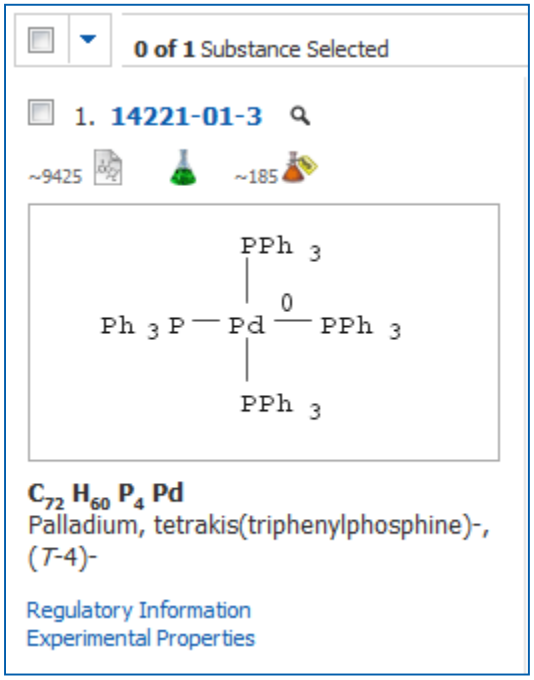
反应结果集庞大时如何限定—反应试剂的限定

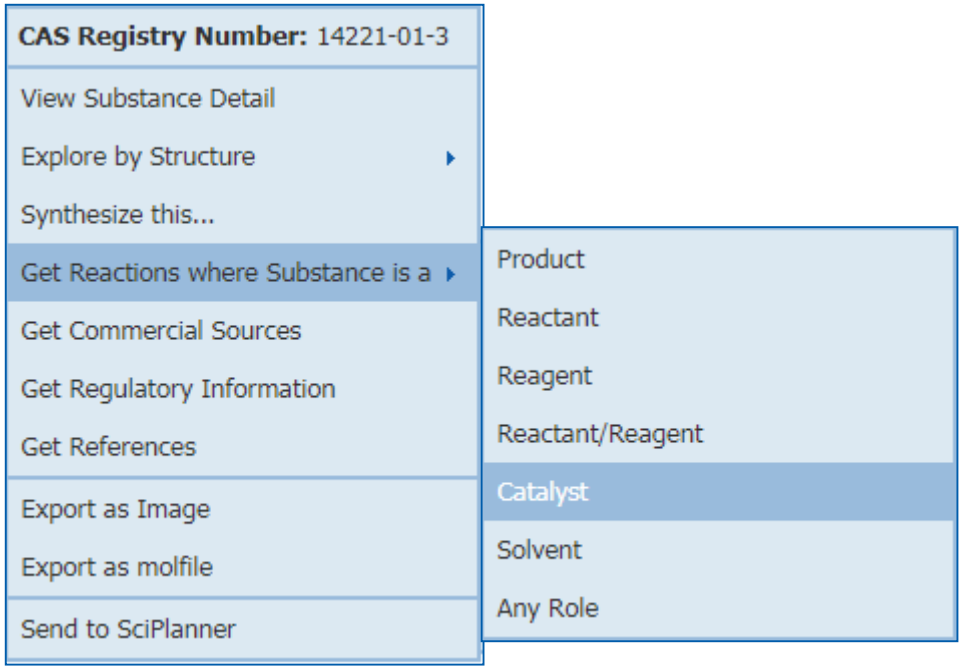


反应结果集庞大时如何限定—催化剂的限定

获得四三苯基磷钯作为催化剂的反应

1. 先获得四三苯基磷钯这个化合物
2. 获得催化剂的反应





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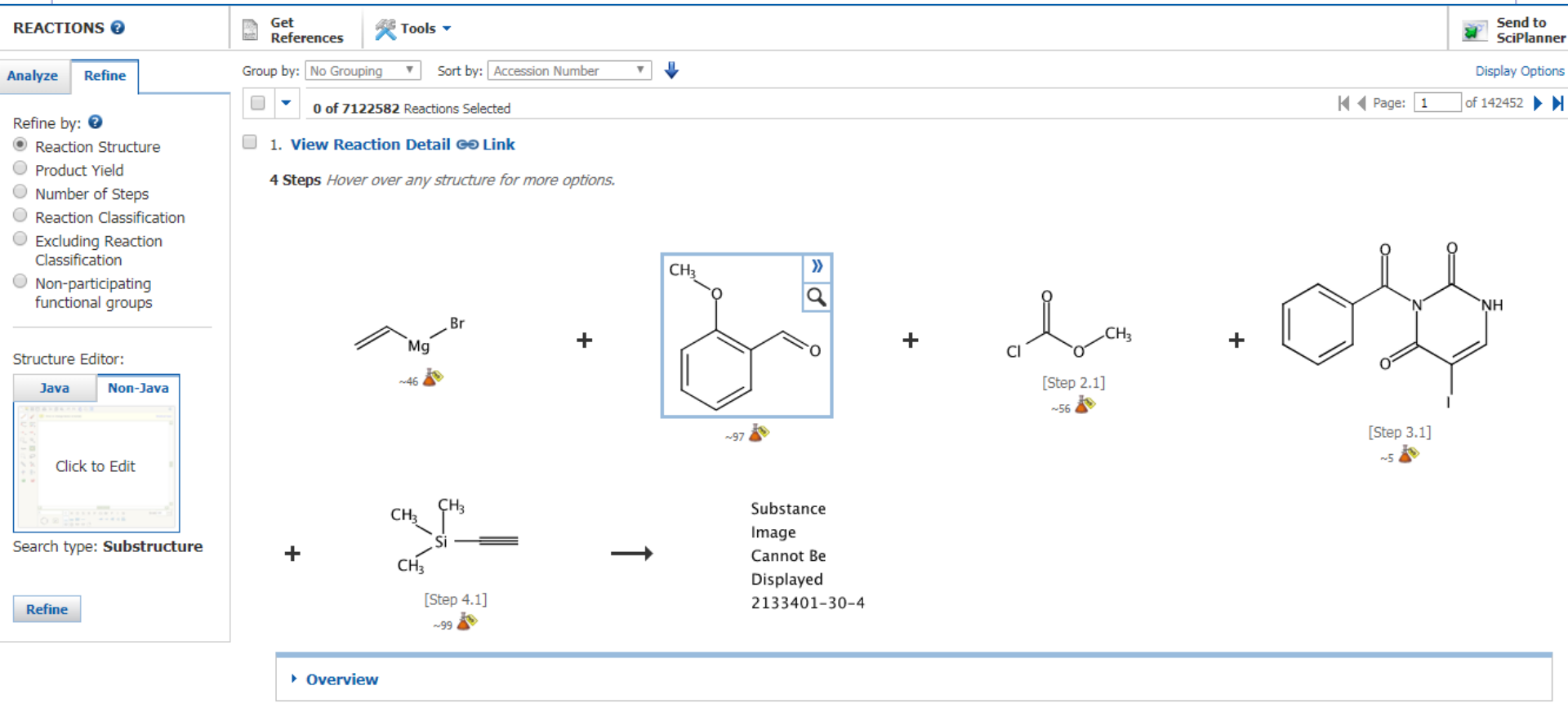
反应结果集庞大时如何限定—催化剂的限定



如有结构，再通过Refine做结构限定

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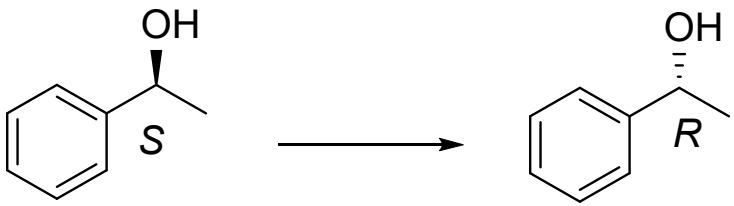
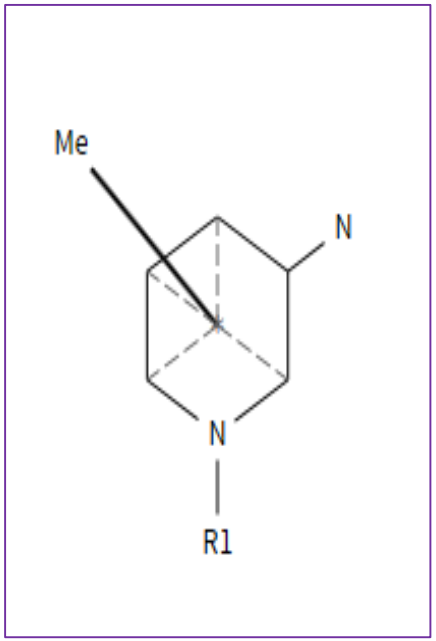
直接检索反应受限的处理

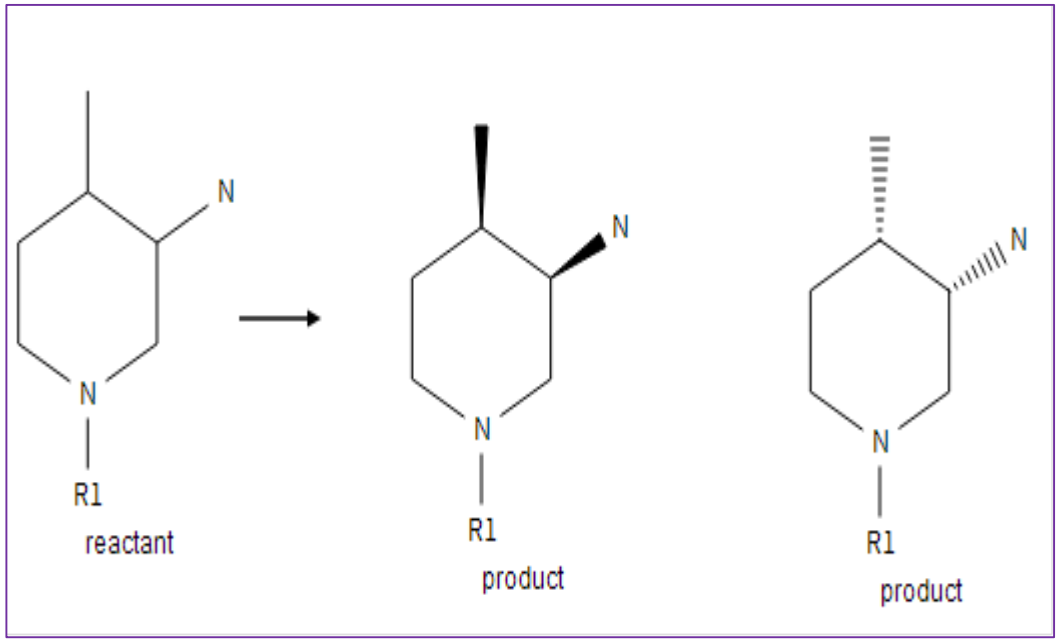
检索如下构型翻转的反应



手性拆分反应

如何检索如下消旋体拆分获得绝对构型的反应？





N-保护的3-氨基-甲基哌啶

举例：R1可以是一些保护基，比如Boc、Cbz等，
甲基可以在吡啶环的2,4,5或6位的任意位置。

手性拆分反应：

- 检索思路1：不考虑物质的手性，快速检索
- 检索思路2：考虑物质的手性，精准检索
- 都是从物质检索出发，然后获取反应，再限定反应物和产物结构

手性拆分反应：检索思路1

- 从产物的反应检索出发

  在反应结构编辑器中，同时绘制两个产物结构（无需画立体构型），选择亚结构反应检索

- 限定反应物

  在反应结果集中，通过Refine: Reaction structure对反应进行限定，所画结构为没有立体构型的反应物

- 获得结果集

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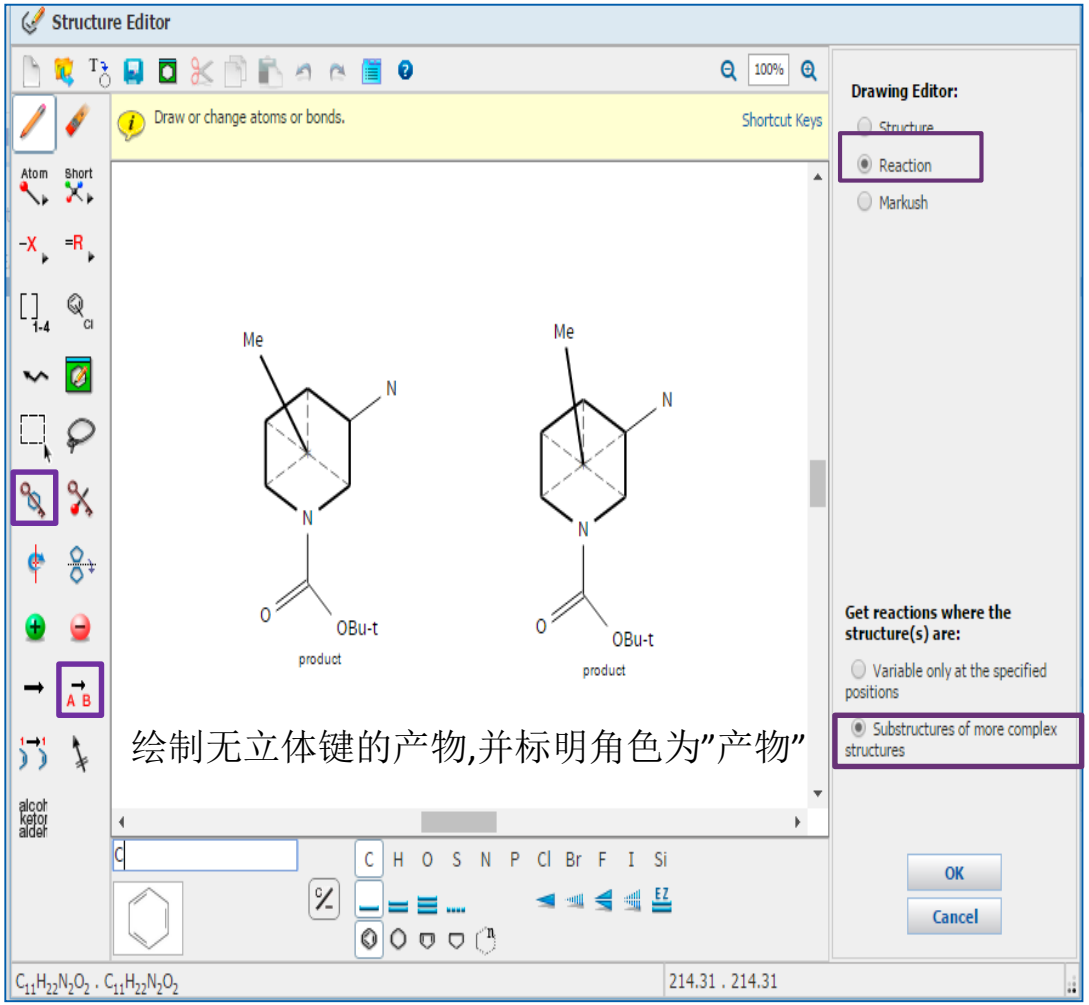
手性拆分反应：从产物的反应检索出发



反应

亚结构反应检索

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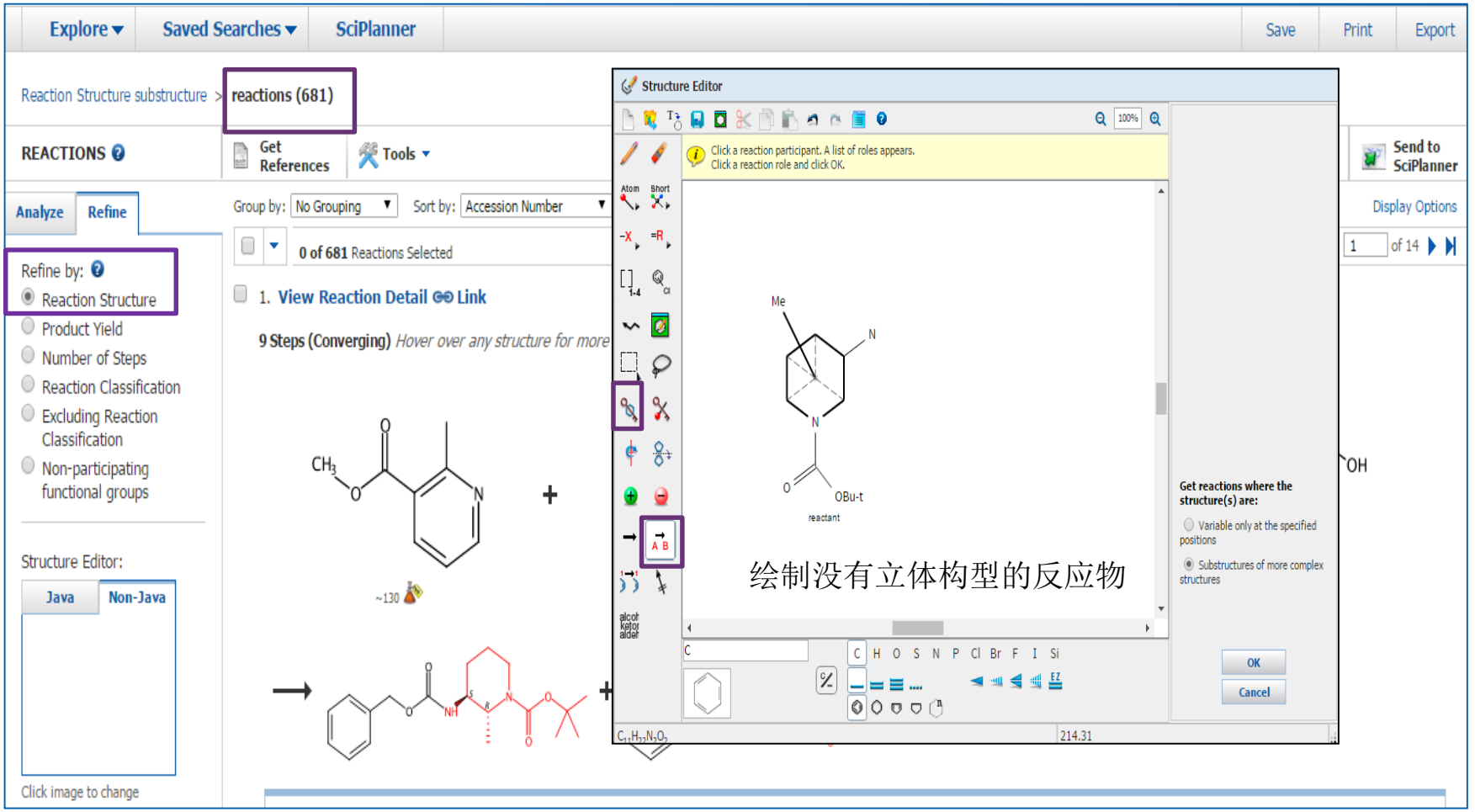
手性拆分反应：限定反应物



在反应结果集中通过Reaction Structure限定反应物

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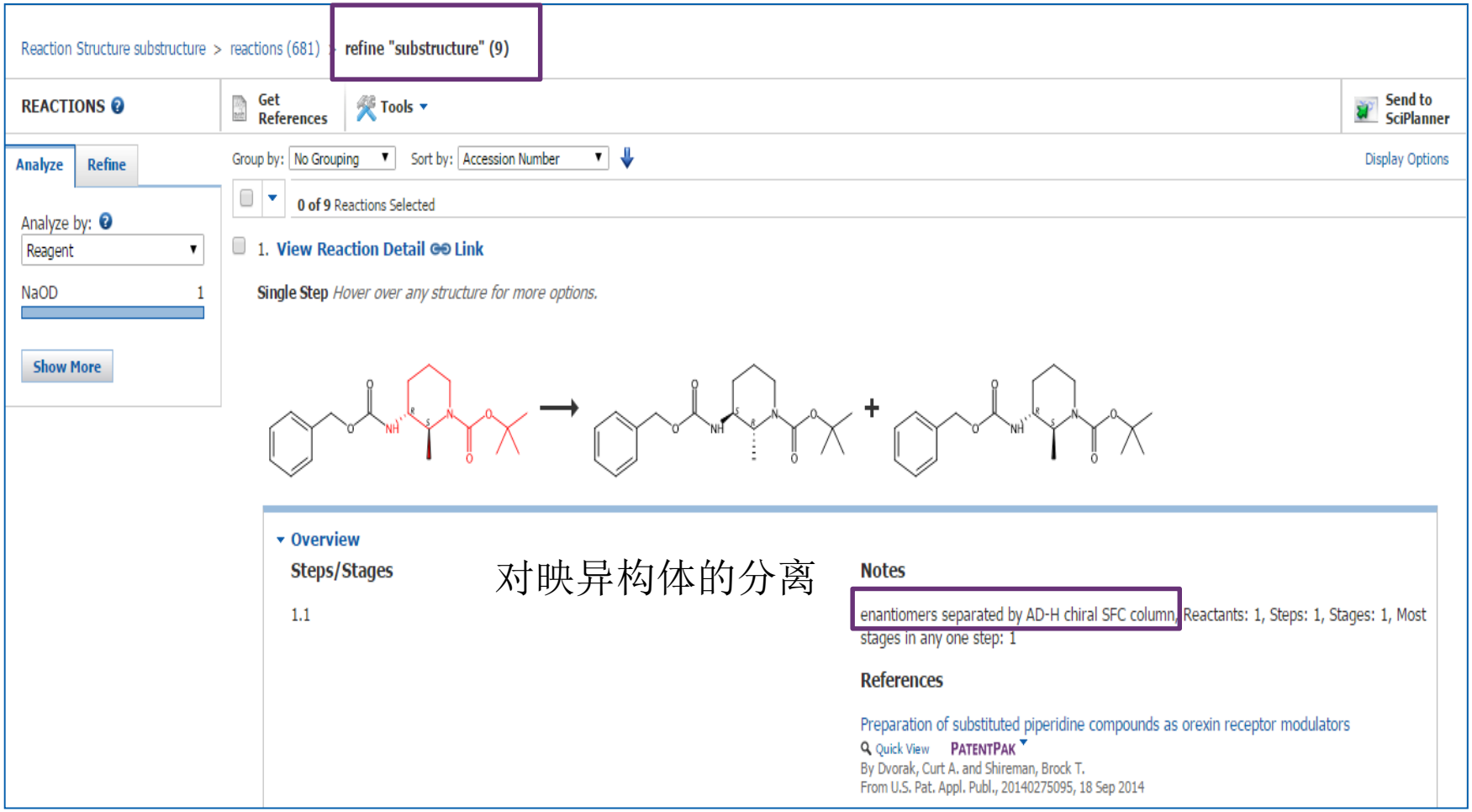
手性拆分反应：获得反应结果集



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检索思路2

- 从物质检索出发，获取反应
  - 反应物的反应：从物质检索出发，获取反应物（挑选没有立体构型的物质结果集）结果集，点击Get Reaction获得反应结果集1
  - 产物的反应：与1采取相同策略（挑选具有绝对立体构型的物质结果集），获得产物的反应结果集2。
- 反应结果集处理
  - 合并反应结果集1与2，取交集获得结果集3
  - 在3的结果集中，用Reaction Structure进行Refine处理：同时绘制两个产物结构（无需画立体构型），并标明角色都为产物
  - 查看反应结果集，可根据需求（如反应步数，产率，反应类型等）限定结果

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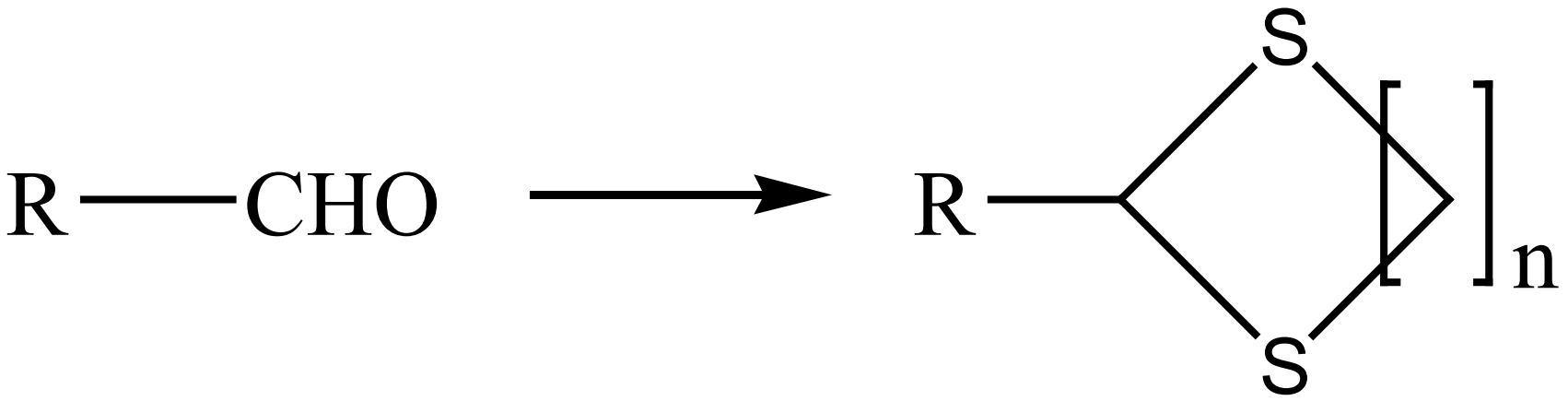
手性拆分反应：

- 检索思路1：不考虑物质的手性，有噪音，但快速获得结果；
- 检索思路2：考虑物质的手性，步骤稍多，但无噪音，精准获得结果；
- 可根据检索要求的复杂程度，灵活地选择检索策略，进行快速或精准的检索。

SciFinder中的反应排除策略

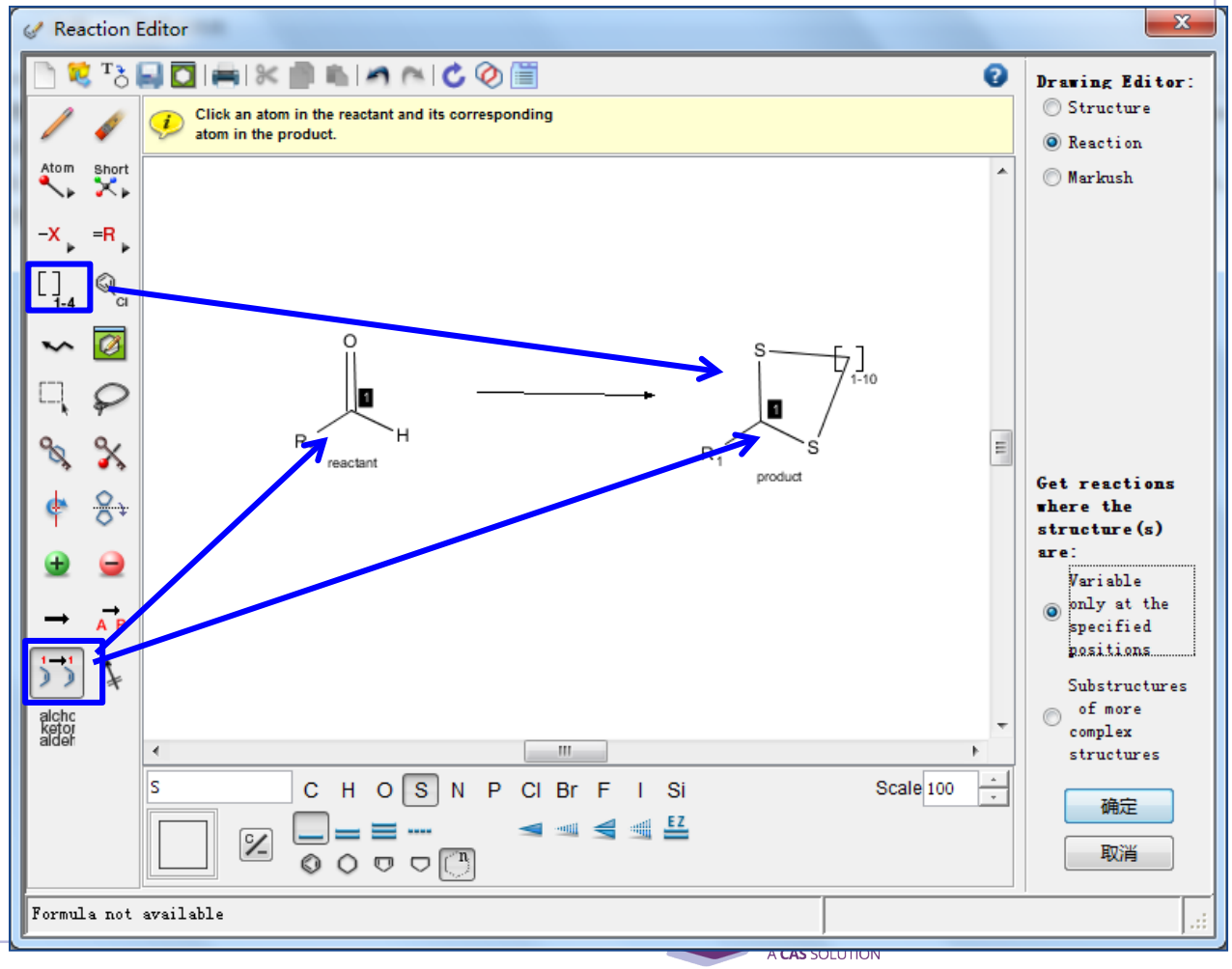
在做以下反应的过程中，不允许使用硫醇作为反应物



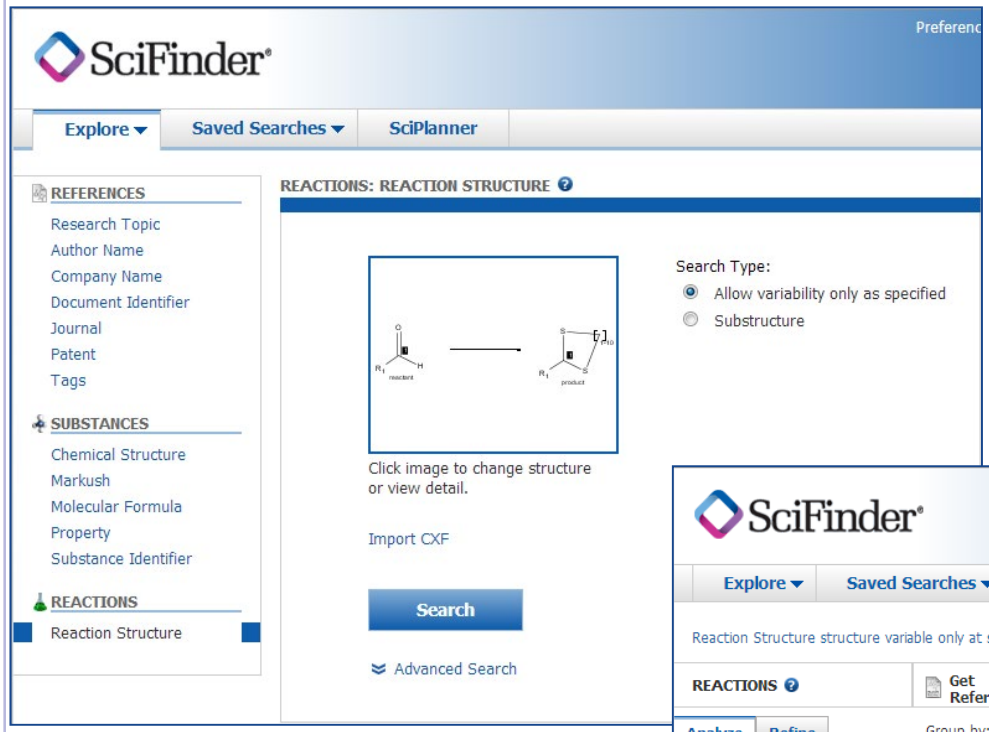
SciFinder®
A CAS SOLUTION

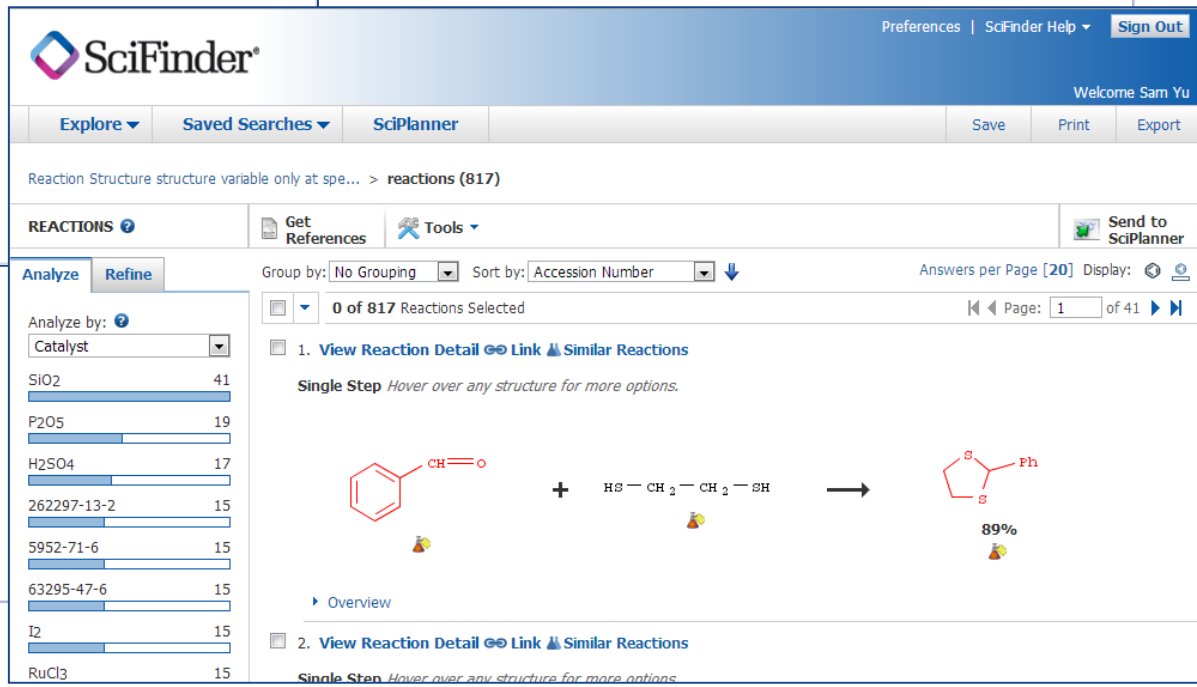
获得全部符合反应变化要求的反应

- 使用R基团定义R1=Ak,Cy
- 使用重复基团定义，定义产物C原子的重复阈值
- 使用原子标记工具，标记成环位置。

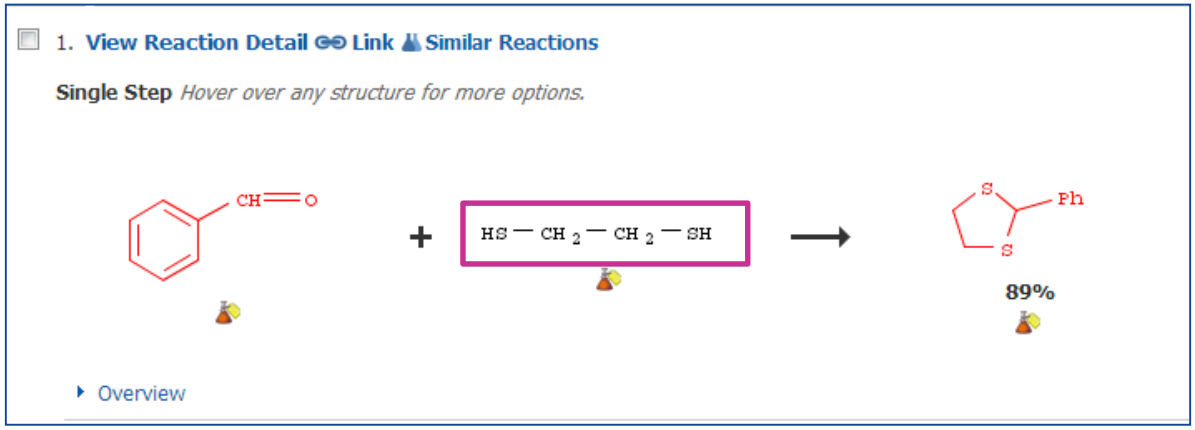


获得全部符合反应变化要求的反应

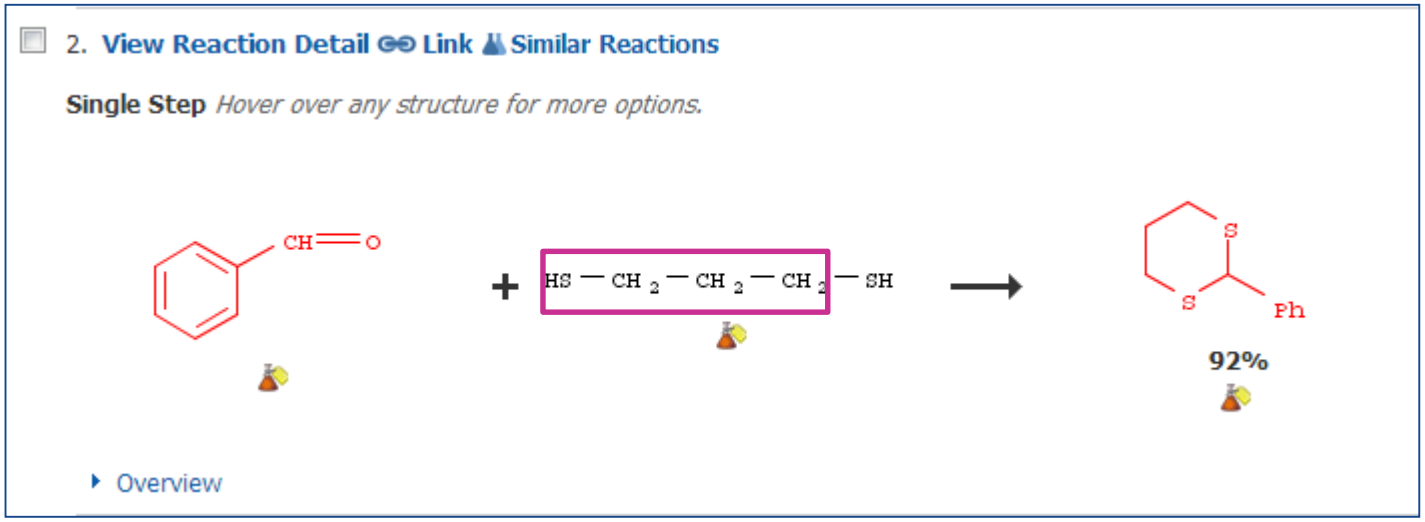




获得全部符合反应变化要求的反应

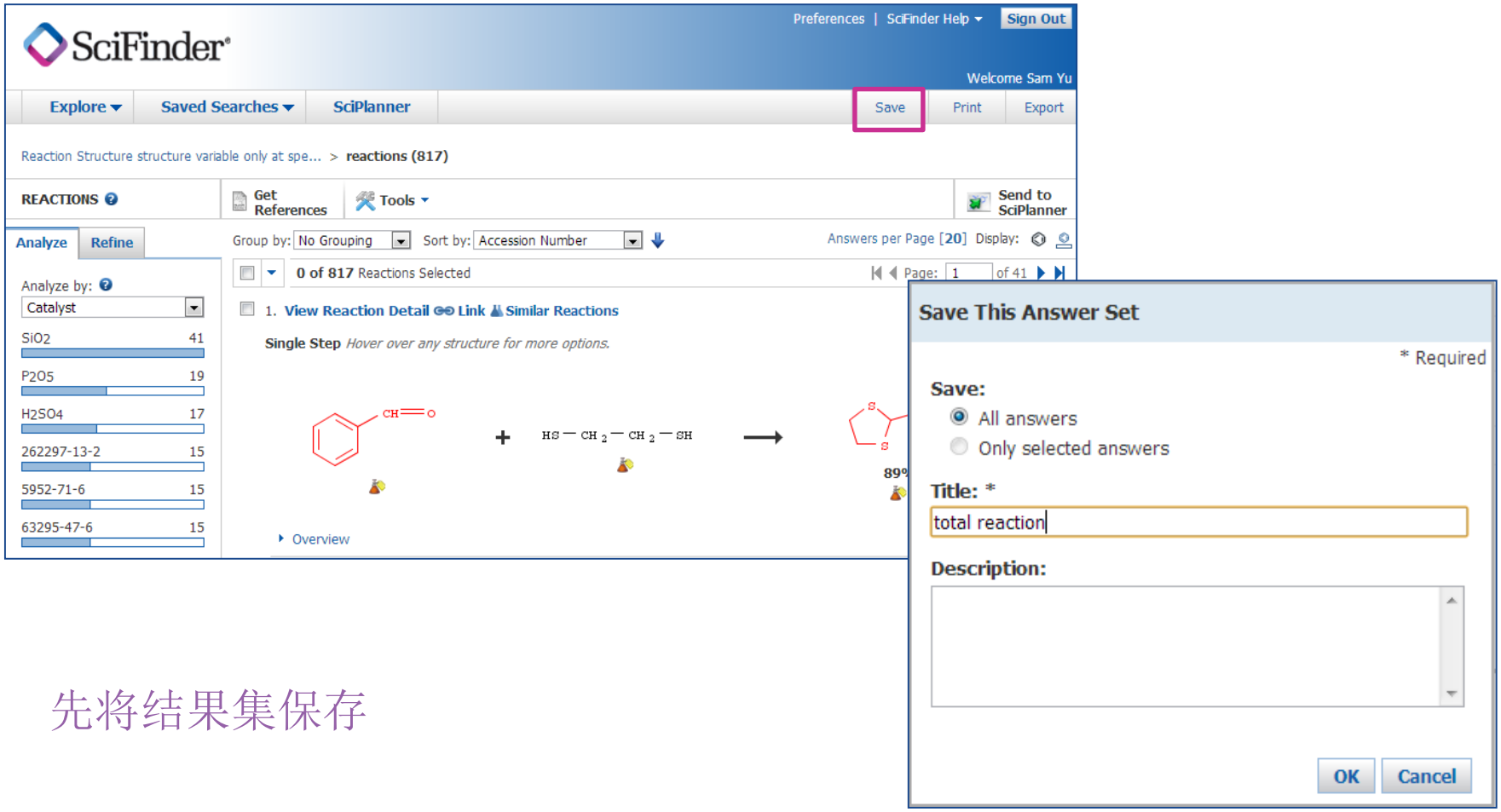


大部分反应都是用硫醇



SciFinder® A CAS SOLUTION

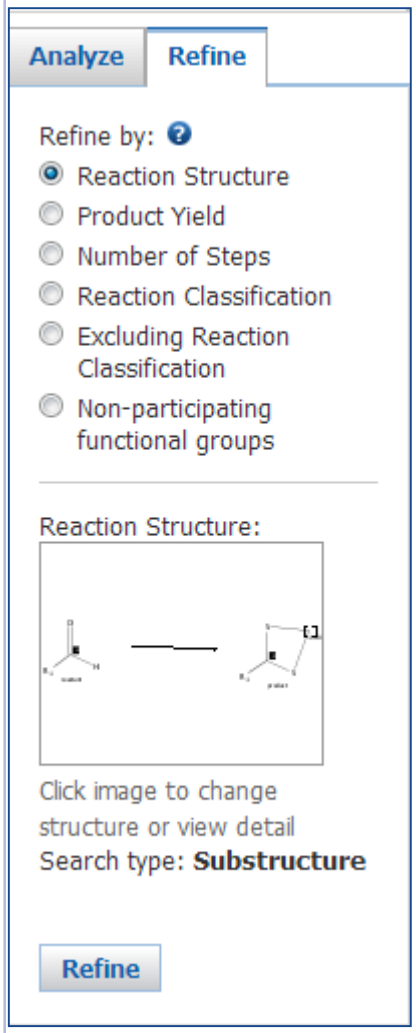
获得全部符合反应变化要求的反应

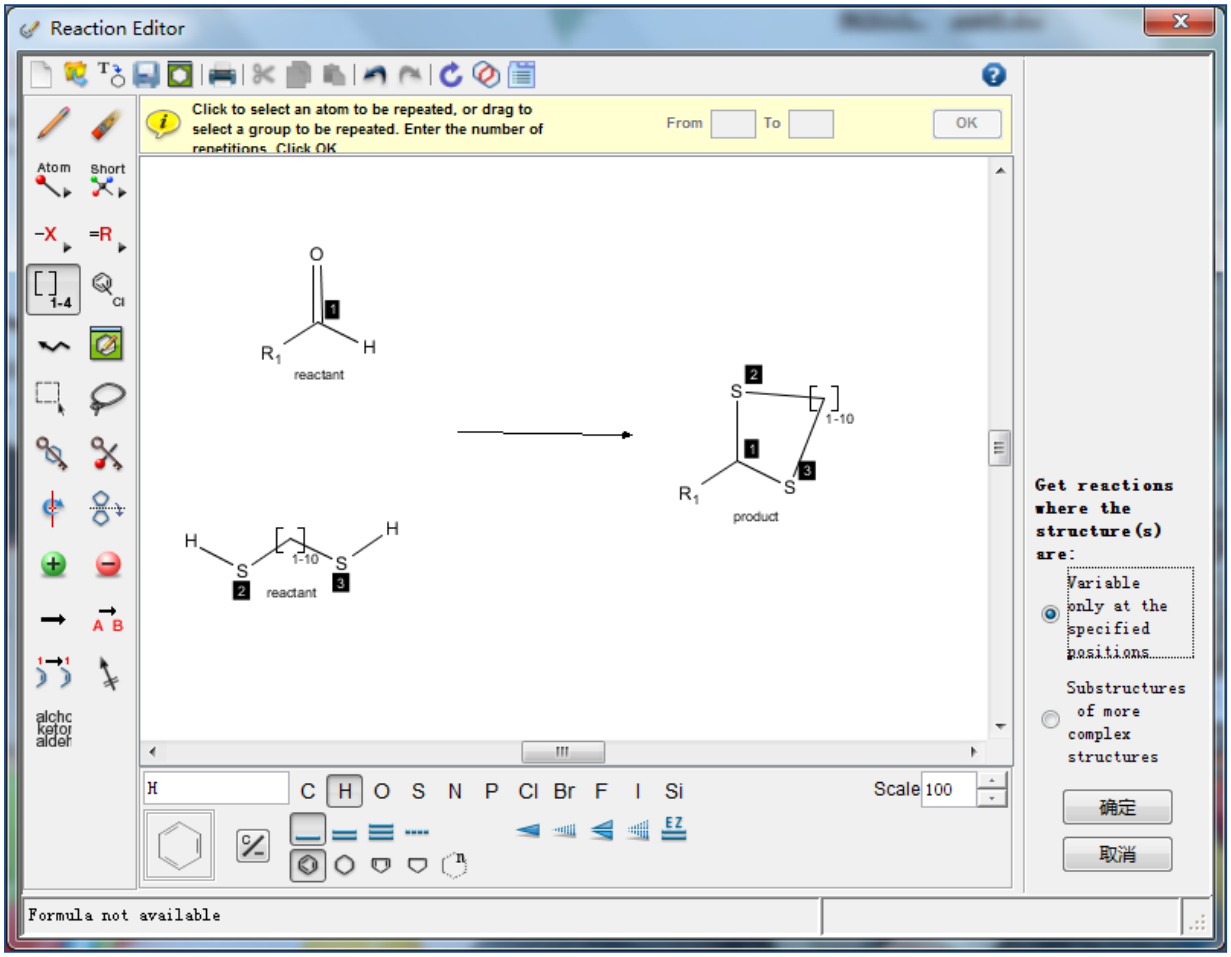


先将结果集保存

SciFinder®
A CAS SOLUTION

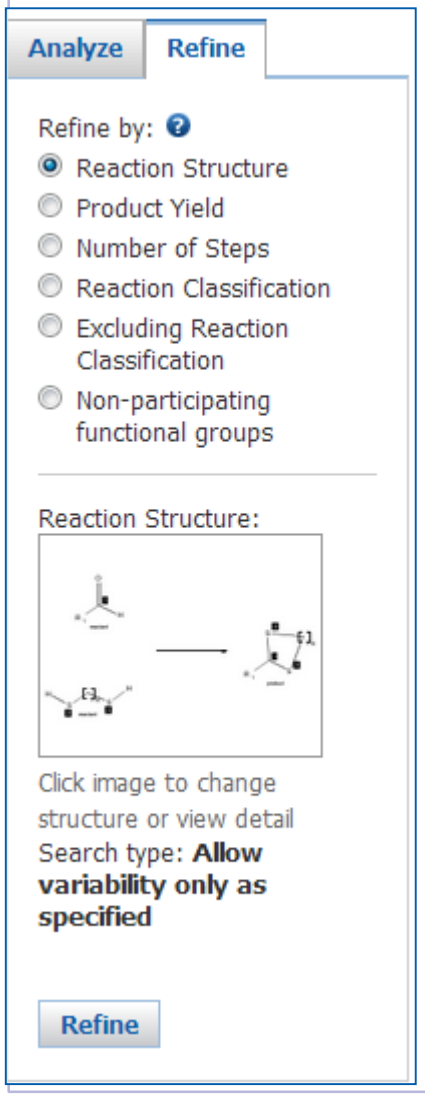
获得需要排除的反应

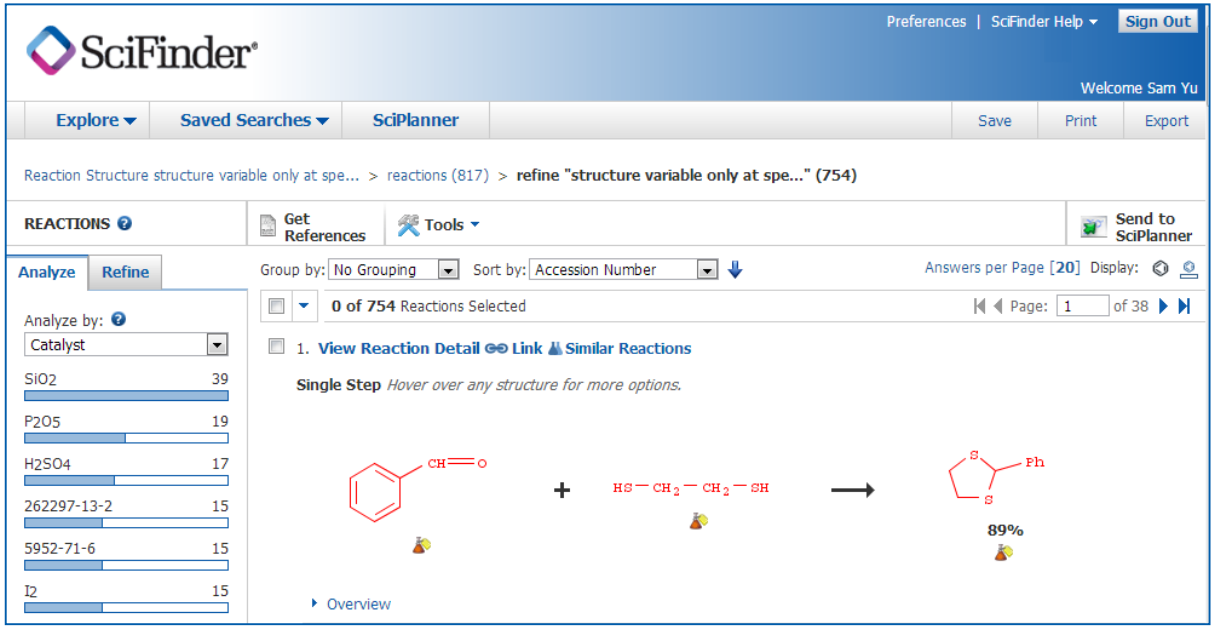




SciFinder® A CAS SOLUTION

获得需要排除的反应





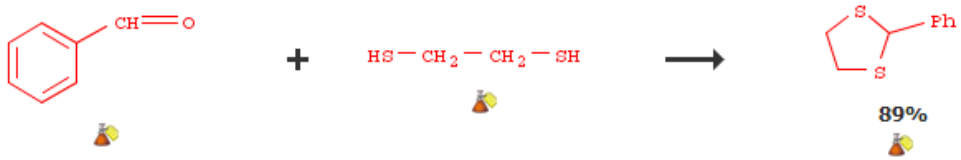
这里限定出来的都是我们不需要的反应

SciFinder® A CAS SOLUTION

获得需要排除的反应

1. View Reaction Detail Link Similar Reactions

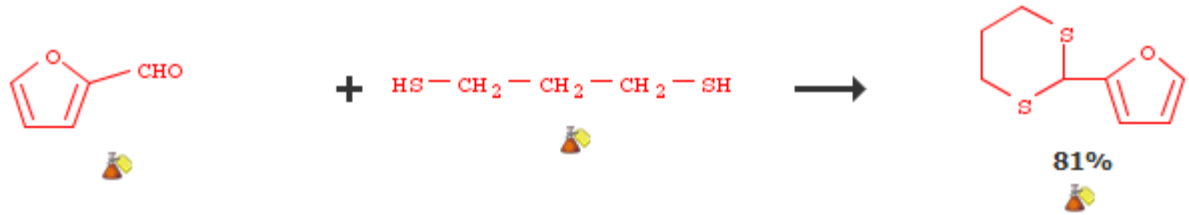
Single Step *Hover over any structure for more options.*



Overview

3. View Reaction Detail Link Similar Reactions

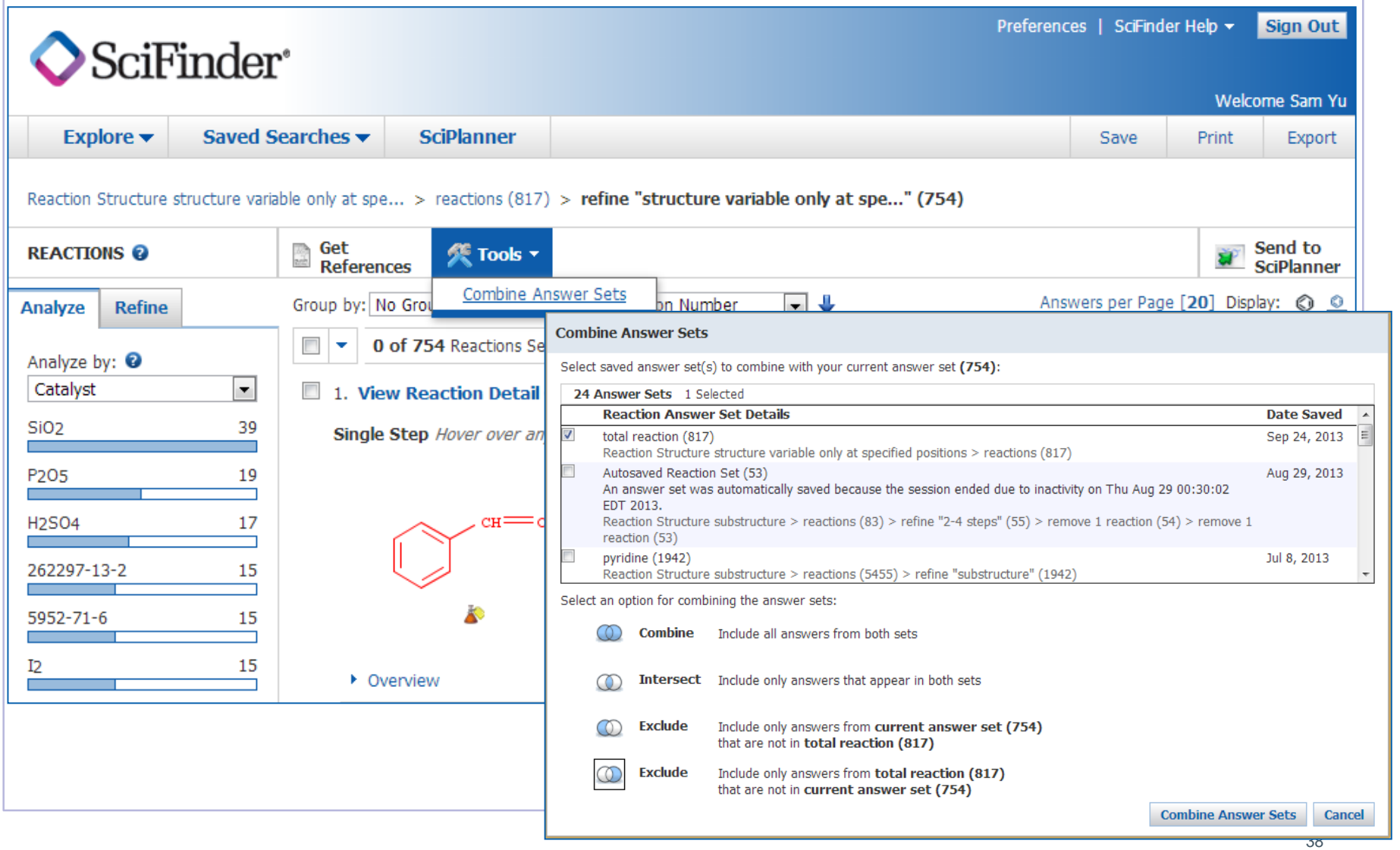
Single Step *Hover over any structure for more options.*



Overview

SciFinder® A CAS SOLUTION

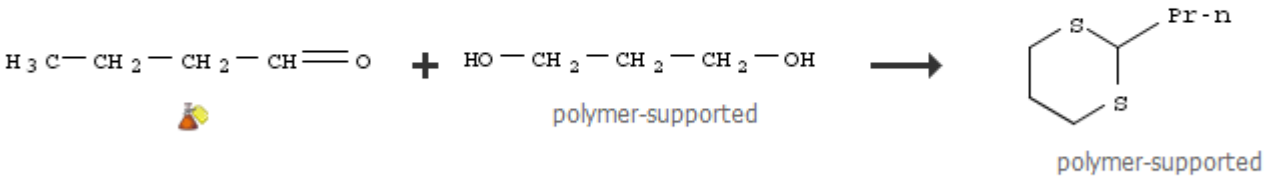
去除不要的反应—Combine



最后的结果

1. View Reaction Detail | Link | Similar Reactions

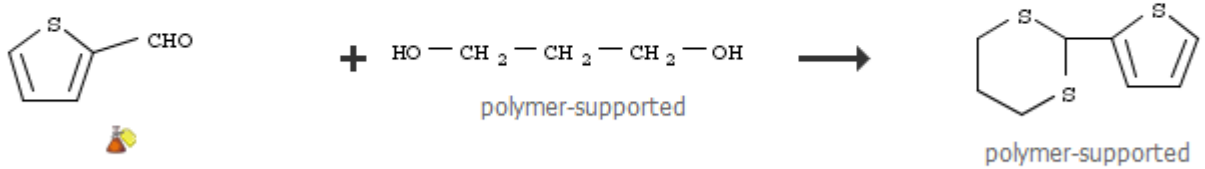
Single Step *Hover over any structure for more options.*



Overview

2. View Reaction Detail | Link | Similar Reactions

Single Step *Hover over any structure for more options.*



Overview

SciFinder® A CAS SOLUTION

新化合物合成路线设计

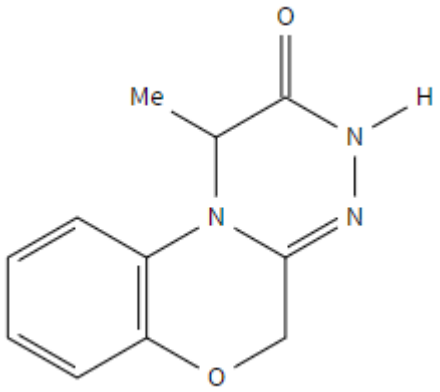
检索思路1：检索结构相似的物质，获得有参考价值的合成路线；

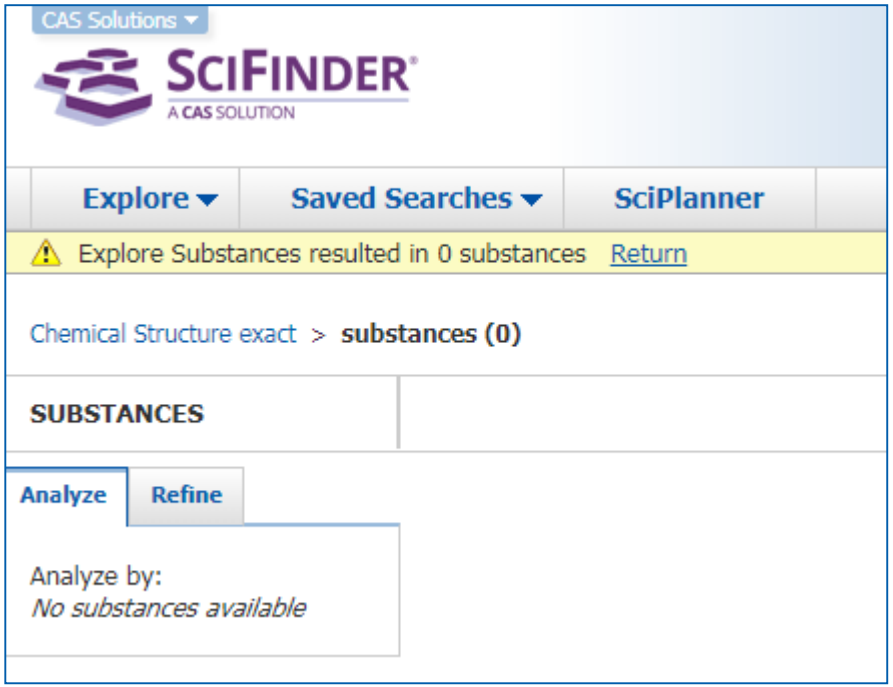
检索思路2：检索通式结构，获得有参考价值的合成路线；

检索思路3：先自行做逆合成反应分析，然后验证相关的反应。

新化合物合成路线设计

检索思路1：检索相似结构，获得有参考价值的合成路线

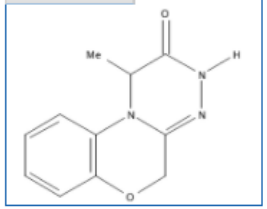




SciFinder
A CAS SOLUTION

Structure Editor:

Java | Non-Java



Click image to change structure or view detail.

Import CXF

Search

Advanced Search | Always Show

Search Type:

- Exact Structure
- Substructure
- Similarity

Show precision

ChemDraw

Launch a SciFi

Select All | Deselect All

1 of 7 Similarity Candidates Selected

	Substances
≥ 99 (most similar)	0
95-98	0
90-94	6
85-89	6
80-84	16
75-79	26
70-74	57
65-69	103
0-64 (least similar)	309

Get Substances

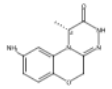
Characteristics

Classes

Studies

0 of 6 Substances Selected

Score: 91
1. **1613721-07-5**

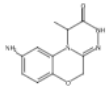


Absolute stereochemistry.

$C_{11}H_{12}N_4O_2$
[1,2,4]Triazino[3,4-*c*][1,4]benzoxazin-2(1*H*)-one, 9-amino-3,5-dihydro-1-methyl-, (1*R*)-

Key Physical Properties

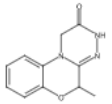
Score: 91
2. **1613722-60-3**



$C_{11}H_{12}N_4O_2$
[1,2,4]Triazino[3,4-*c*][1,4]benzoxazin-2(1*H*)-one, 9-amino-3,5-dihydro-1-methyl-

Key Physical Properties

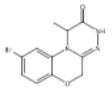
Score: 90
3. **139605-55-3**



$C_{11}H_{11}N_3O_2$
[1,2,4]Triazino[3,4-*c*][1,4]benzoxazin-2(1*H*)-one, 3,5-dihydro-5-methyl-

Key Physical Properties

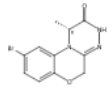
Score: 90
4. **1613721-69-9**



$C_{11}H_{10}BrN_3O_2$
[1,2,4]Triazino[3,4-*c*][1,4]benzoxazin-2(1*H*)-one, 9-bromo-3,5-dihydro-1-methyl-

Key Physical Properties

Score: 90
5. **1613721-93-9**

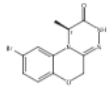


Rotation (-).,Absolute stereochemistry.

$C_{11}H_{10}BrN_3O_2$
[1,2,4]Triazino[3,4-*c*][1,4]benzoxazin-2(1*H*)-one, 9-bromo-3,5-dihydro-1-methyl-, (1*R*)-

Key Physical Properties

Score: 90
6. **1613722-23-8**



Absolute stereochemistry.

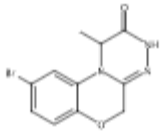
$C_{11}H_{10}BrN_3O_2$
[1,2,4]Triazino[3,4-*c*][1,4]benzoxazin-2(1*H*)-one, 9-bromo-3,5-dihydro-1-methyl-, (1*S*)-

Key Physical Properties

Score: 90

4. **1613721-69-9**

~1 ~2



CAS Registry Number: 1613721-69-9

View Substance Detail
Explore by Structure
Synthesize this...
Get Reactions where Substance is a
Get Commercial Sources
Get Regulatory Information
Get References
Export as Image
Export as molfile
Send to SciPlanner

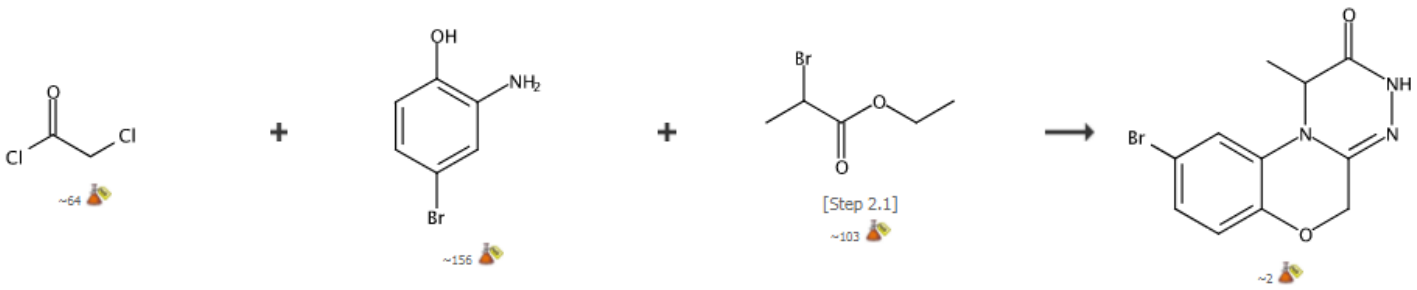
$C_{11}H_{10}BrN_3O_2$

[1,2,4]Triazino[3,4-*c*][1,4]benzoxazin-2(1*H*)-one, 9-bromo-3,5-dihydro-1-methyl-

Key Physical Properties

1. **View Reaction Detail** Link

4 Steps *Hover over any structure for more options.*



Overview

Steps/Stages

1.1 R:$NaHCO_3$, S:H_2O, S:$(CH_2OMe)_2$, 0°C; 0.5 h, 15°C; overnight, 80°C; 80°C → rt
1.2 R:H_2O
2.1 R:K_2CO_3, S:Me_2CO, 5 h, 70°C
3.1 R:Lawesson's reagent, S:PhMe, 3 h, 120°C
4.1 R:N_2H_4-H_2O, S:EtOH, overnight, rt

Notes

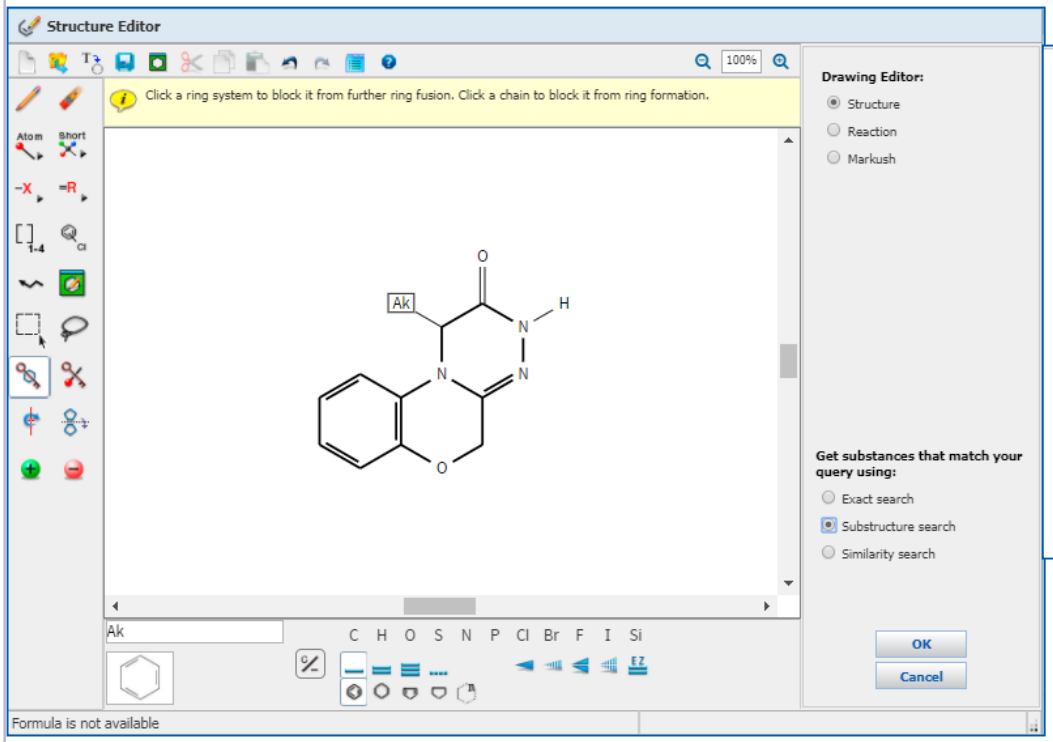
1) alternate reaction conditions gave lower yield, Reactants: 3, Reagents: 5, Solvents: 5, Steps: 4, Stages: 5, Most stages in any one step: 2

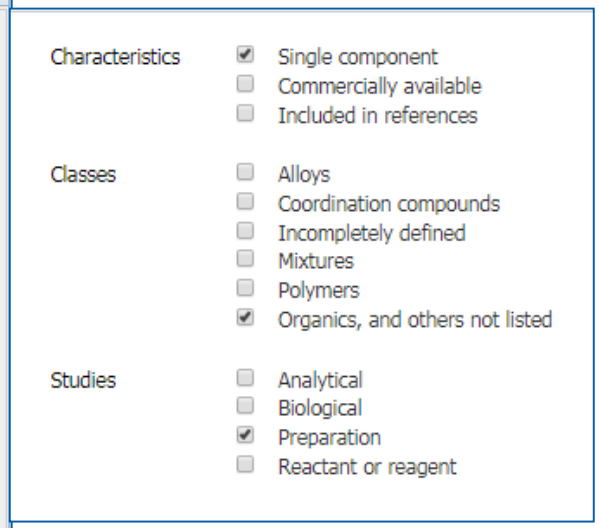
References

Triazinone compounds as PKC kinase inhibitors and their preparation
Quick View PATENTPAK
By George, Dawn M. et al
From PCT Int. Appl., 2014089904, 19 Jun 2014

新化合物合成路线设计

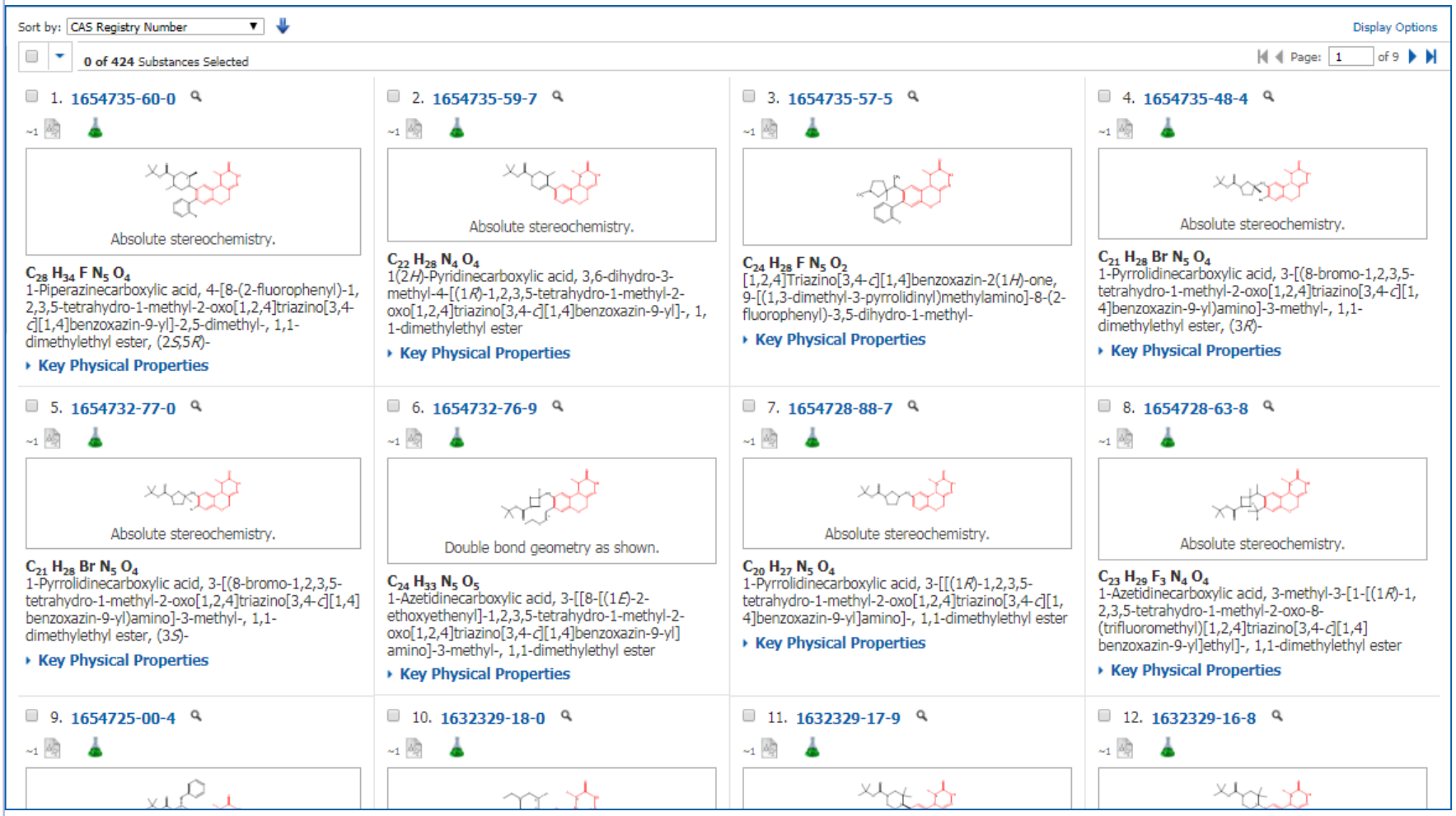
检索思路2：检索通式结构，获得有参考价值的合成路线





SciFinder® A CAS SOLUTION

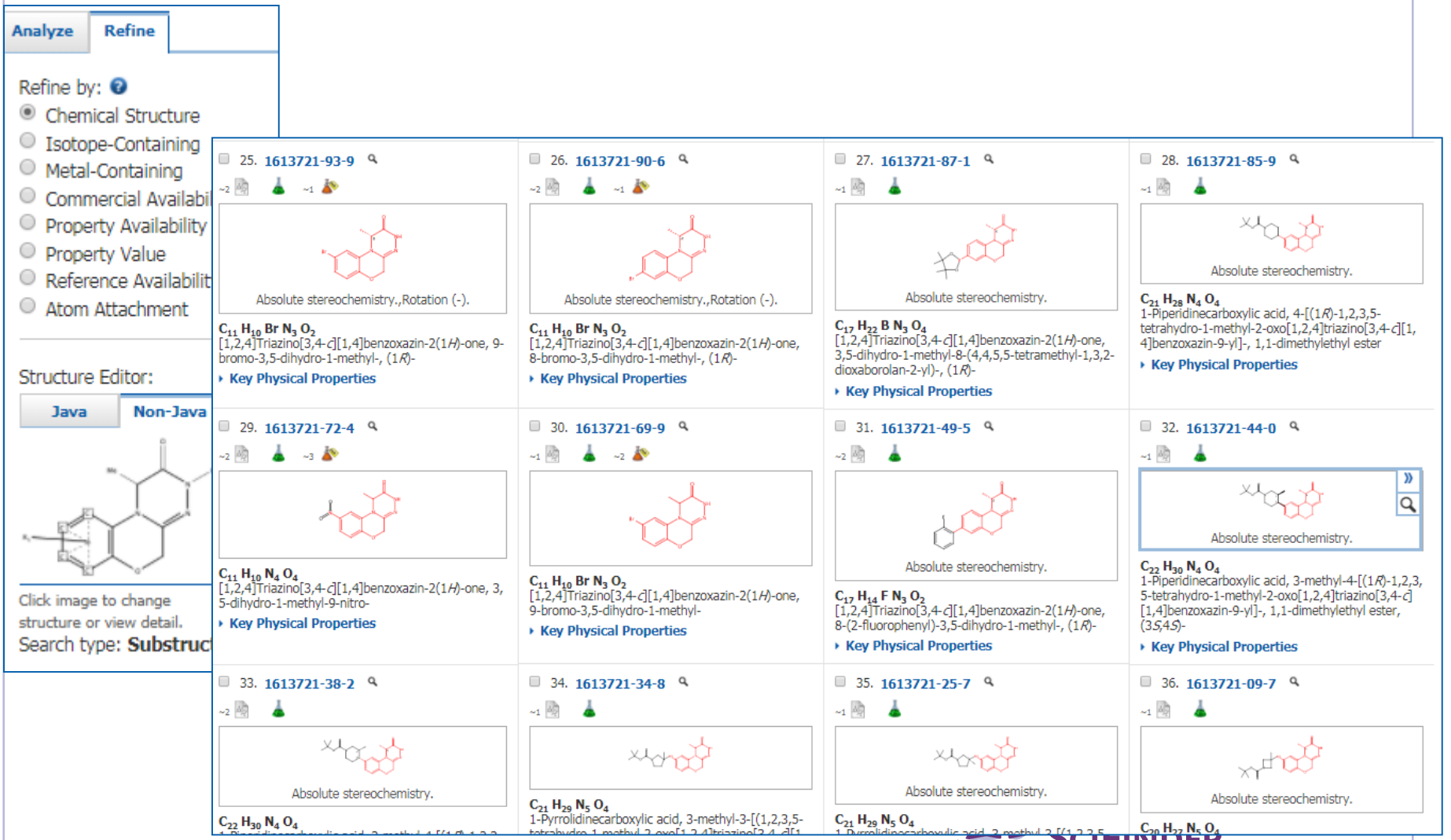
新化合物合成路线设计



筛选结构相近的物质

SciFinder® A CAS SOLUTION

新化合物合成路线设计



SCIFINDER
A CAS SOLUTION

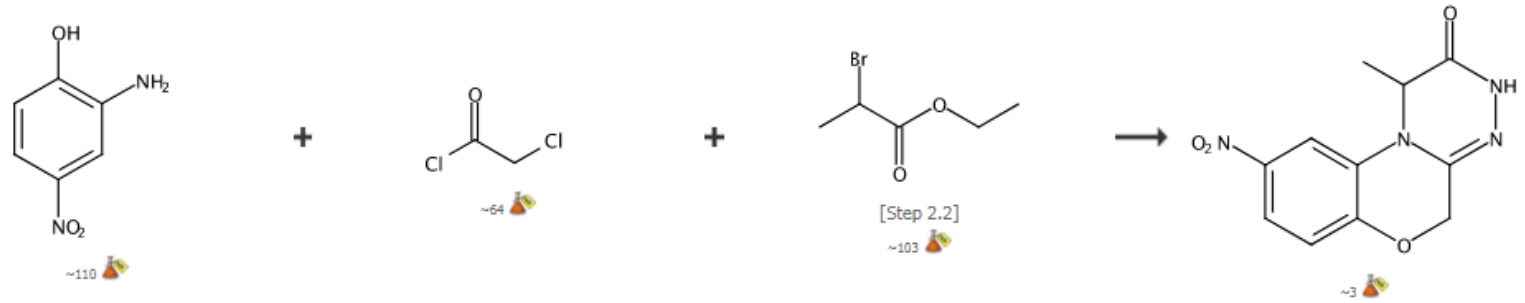
新化合物合成路线设计

Group by: No Grouping ▼ Sort by: Accession Number ▼ Display Options

0 of 8 Reactions Selected

1. View Reaction Detail Link

4 Steps *Hover over any structure for more options.*



▸ **Overview**

▸ **Experimental Procedure**

▾ **METHODSNOW™**

Procedure

1. Add 2-chloroacetyl chloride (83.6 mL, 1110 mmol) dropwise to a solution of 2-amino-4-nitrophenol (114.0 g, 740 mmol) , K_2CO_3 (306.2 g, 2220 mmol) and tetrabutyl ammonium bromide (23.8 g, 74 mmol) in acetonitrile (1.50 L) at 0 °C.
2. Heat the reaction mixture at 80 °C for 1 h.

View more...

Available Experimental Data

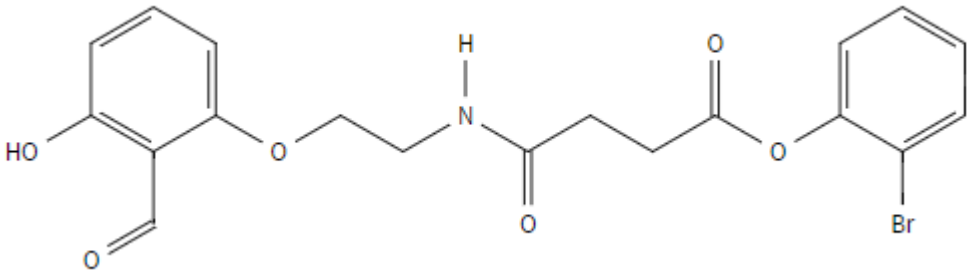
^{1}H NMR, R_f, State

View with MethodsNow

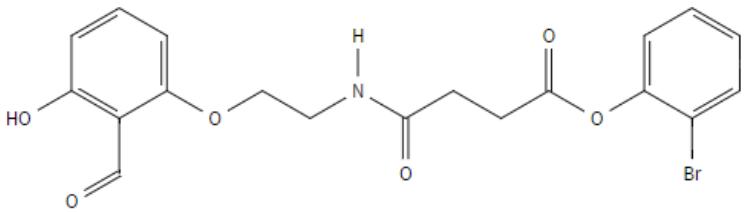
SCIFINDER® A CAS SOLUTION

新化合物合成路线设计

检索思路3：先自行做逆合成反应分析，然后检索相关的反应



逆合成反应分析



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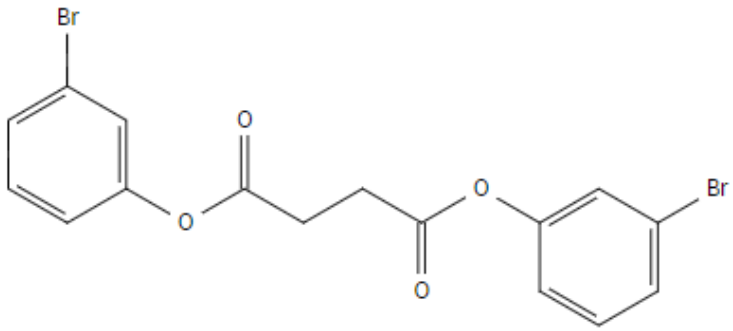

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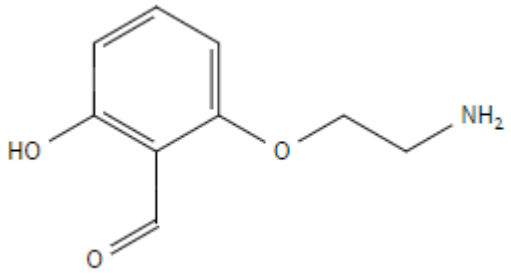

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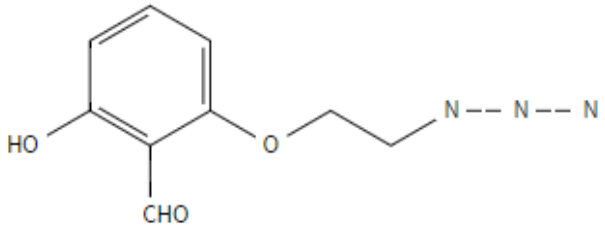
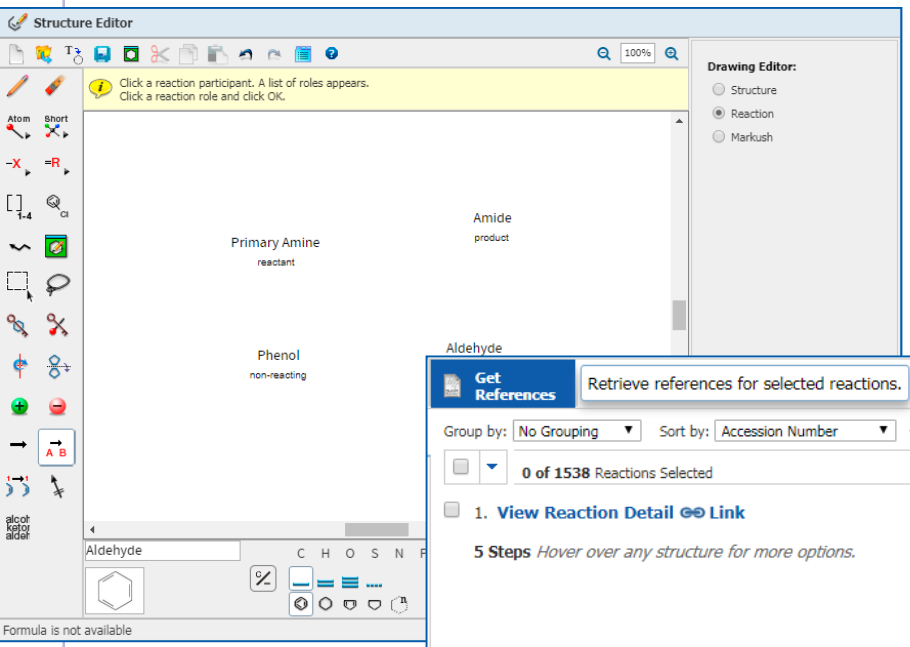
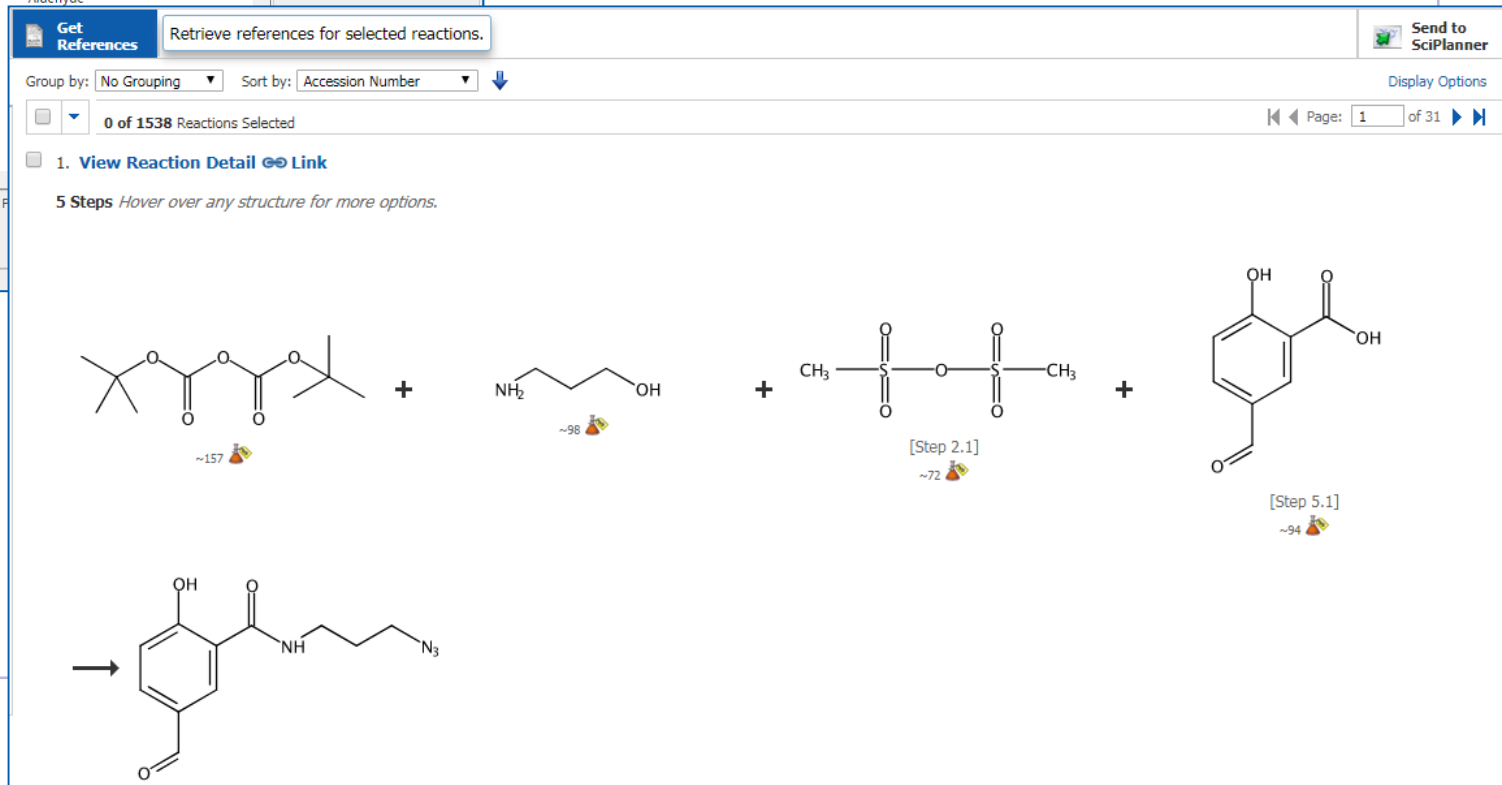


SciFinder® A CAS SOLUTION

新化合物合成路线设计

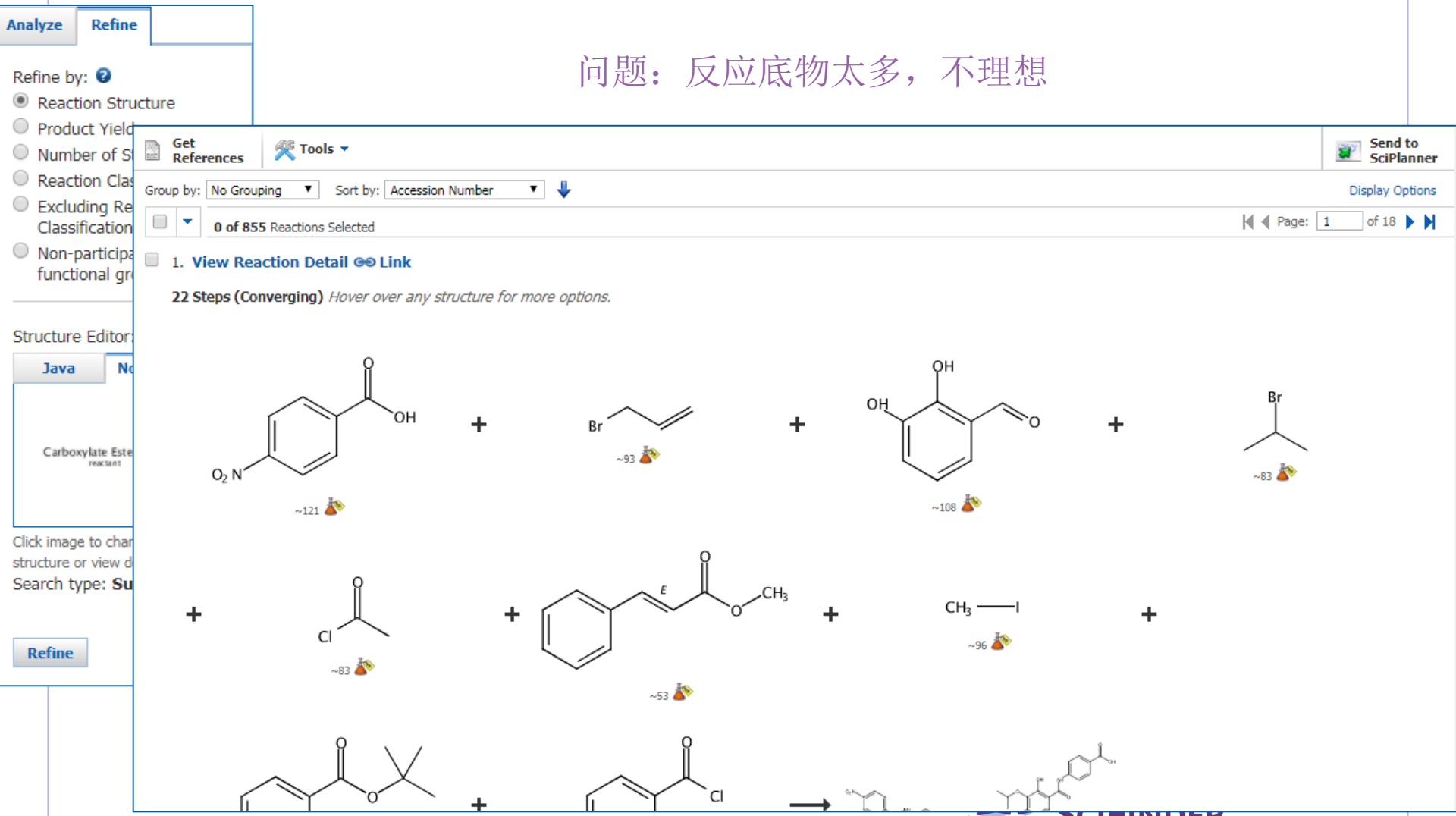
反应1





新化合物合成路线设计

问题：反应底物太多，不理想



新化合物合成路线设计

进一步筛选

Analyze | Refine

Refine by:

- Reaction Structure
- Product Yield
- Number of Step
- Reaction Classif
- Excluding React Classification
- Non-participatin functional group

Number of Steps: 1-2

Examples: *1, 1 - 3, 1 -*

Refine

Get References | Tools | Send to SciPlanner

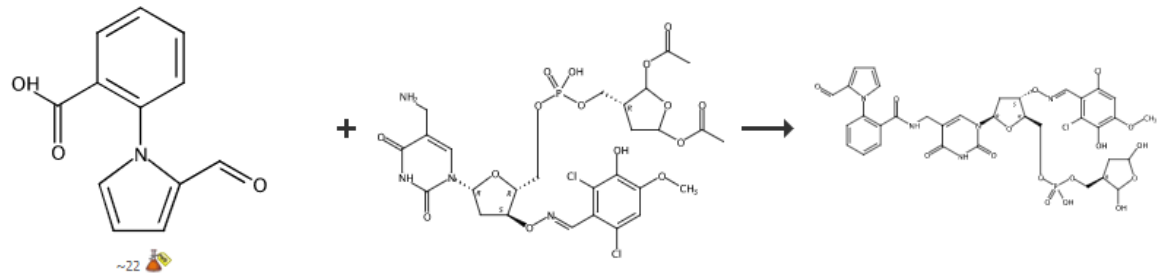
Group by: No Grouping | Sort by: Accession Number | Display Options

0 of 66 Reactions Selected | Page: 1 of 2

1. **View Reaction Detail Link**

2 Steps *Hover over any structure for more options.*

酰化剂为羧酸，不符合要求

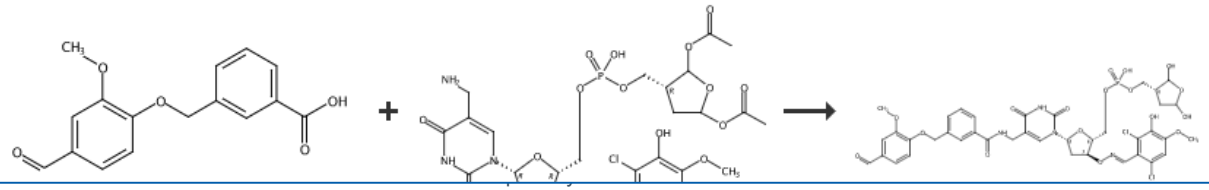


~22

Overview

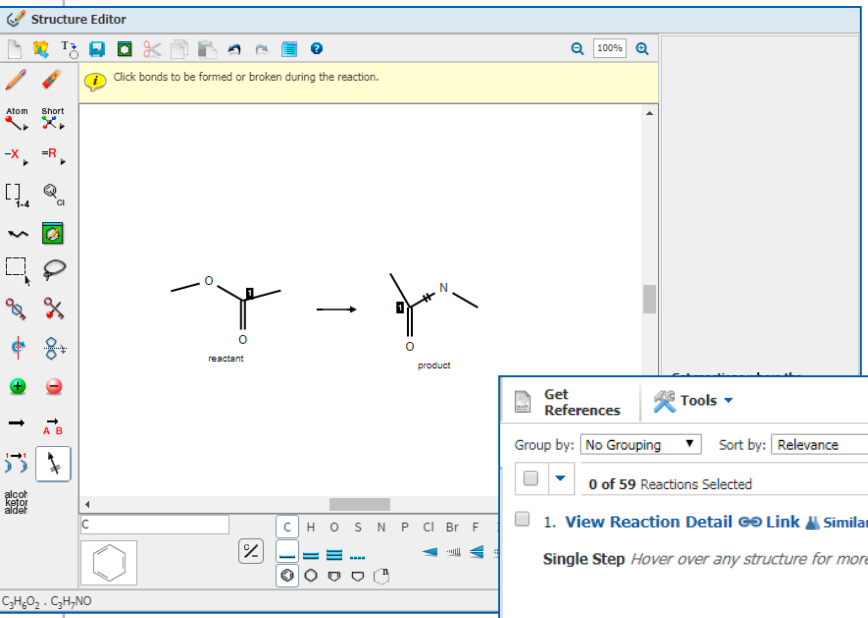
2. **View Reaction Detail Link**

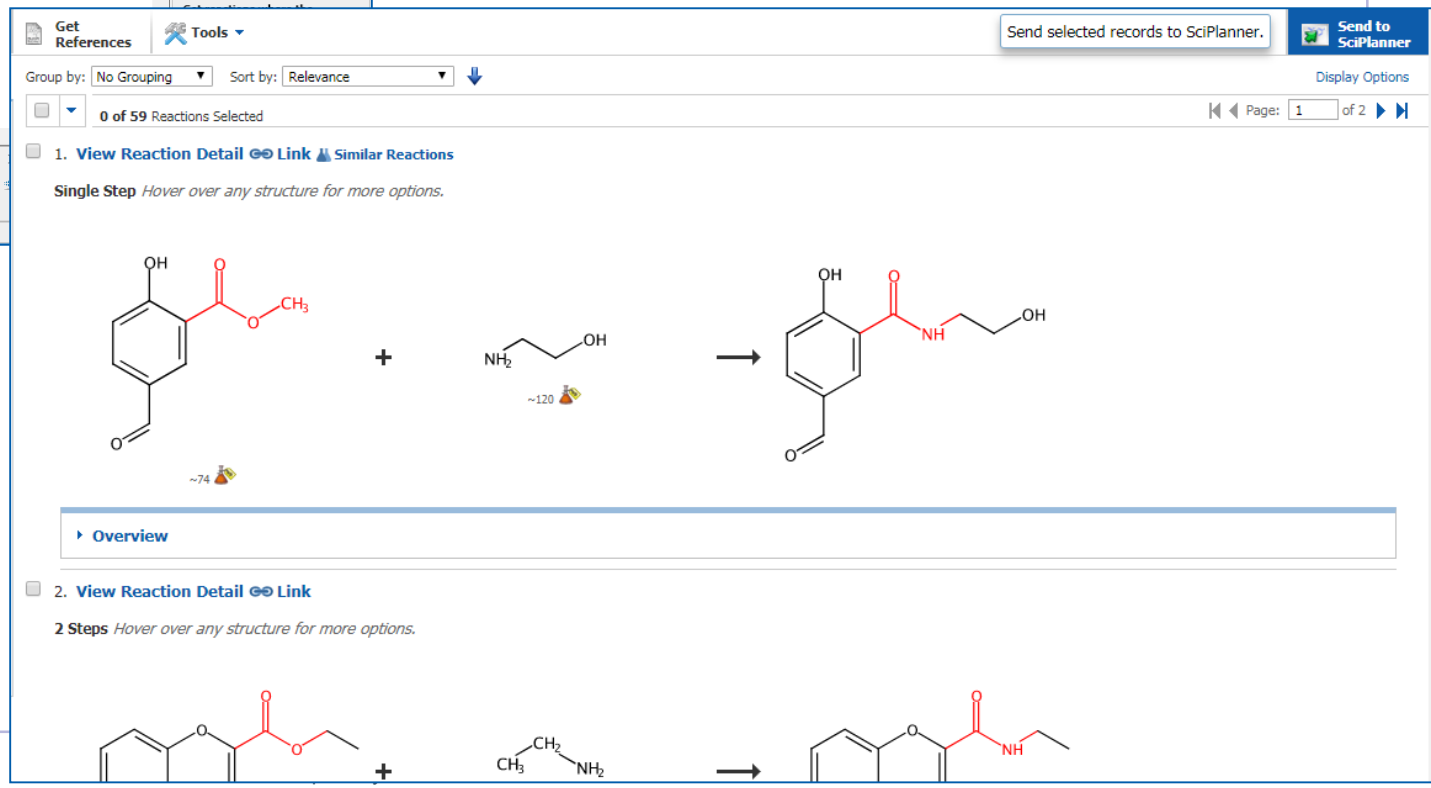
2 Steps *Hover over any structure for more options.*



新化合物合成路线设计

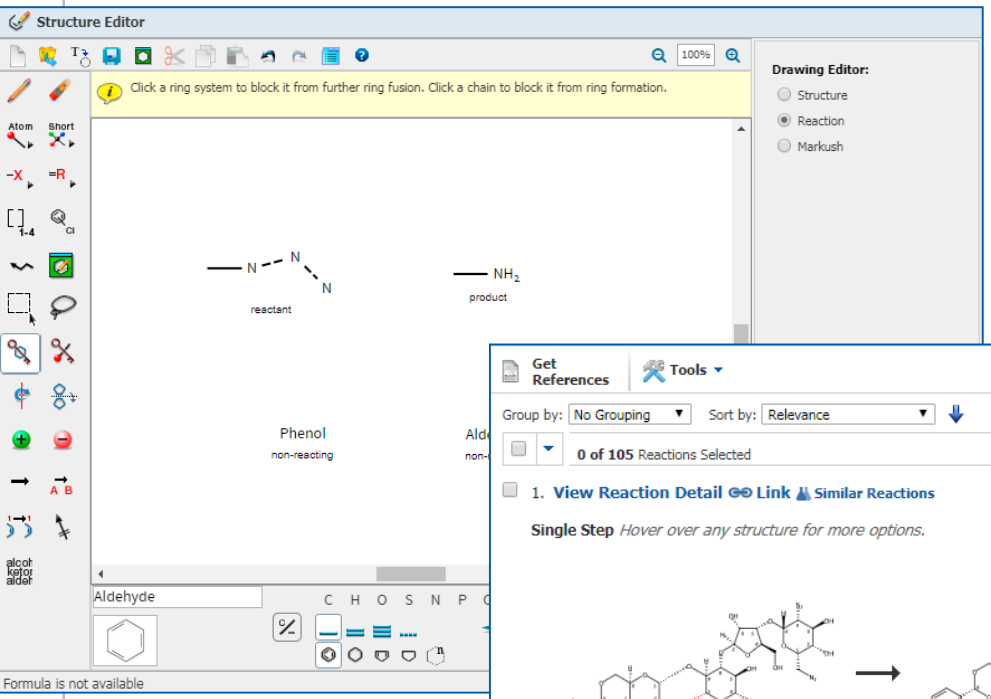
进一步限定用酯做酰化剂的反应

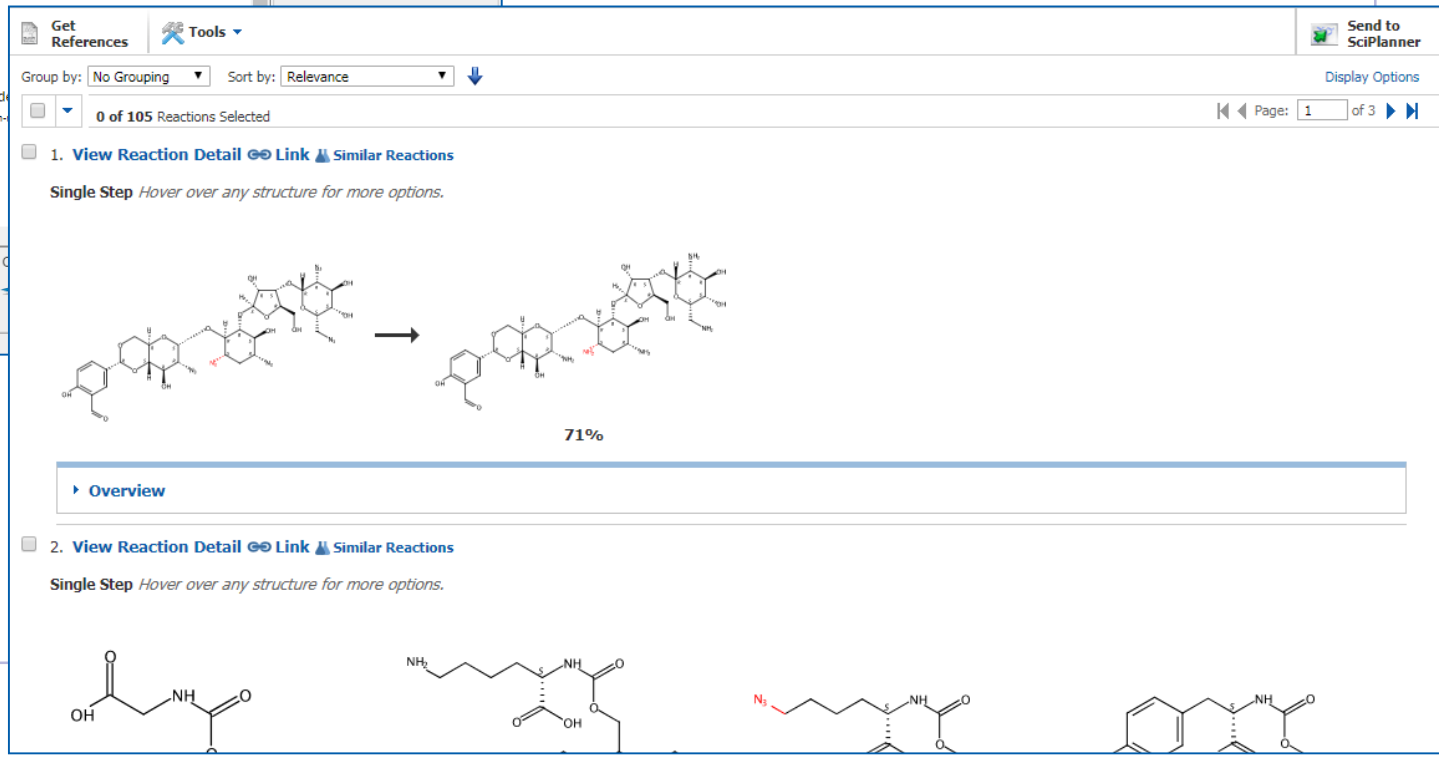




新化合物合成路线设计

反应2





案例分析

检索要求：

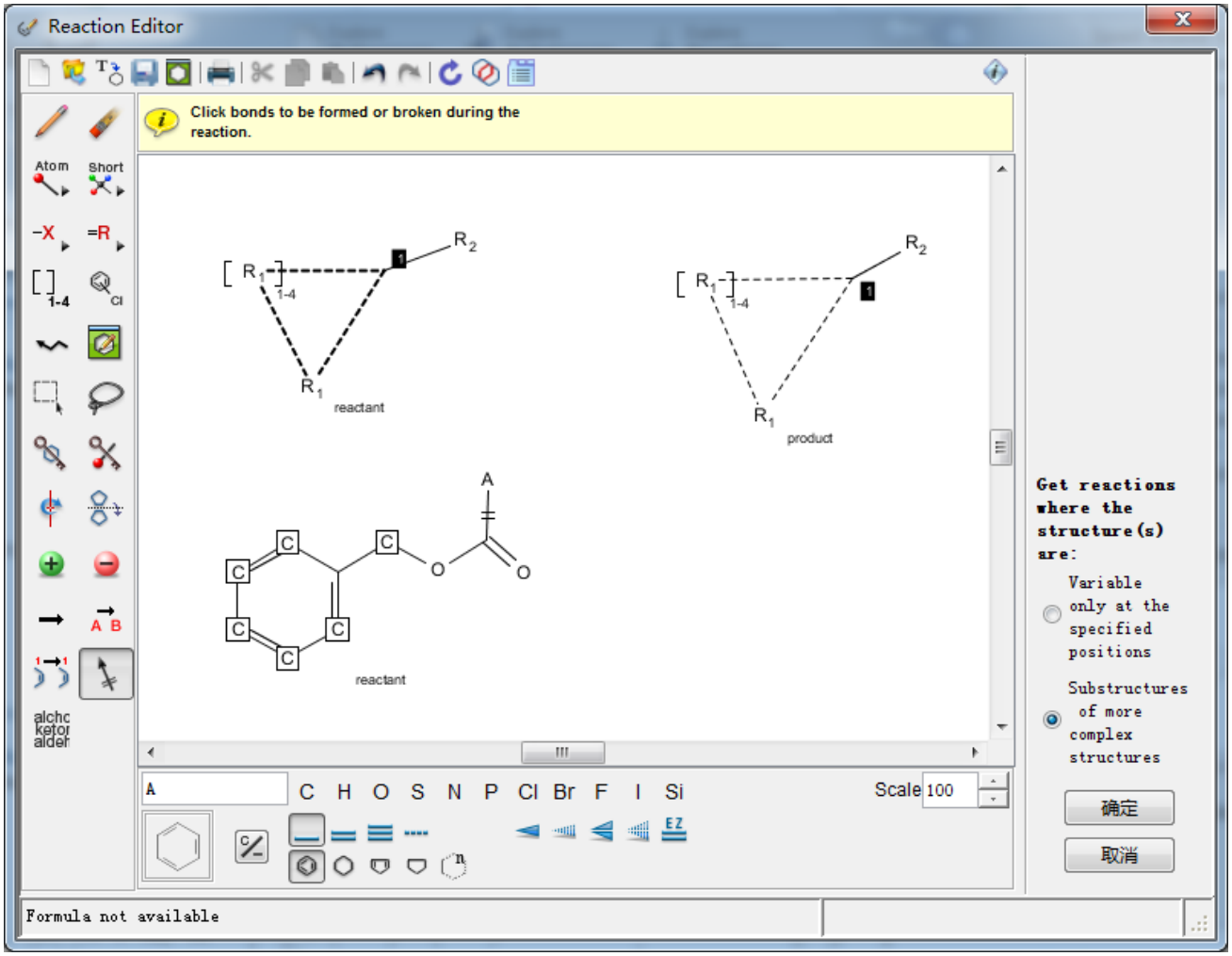
- 某个结构中包含环和Cbz，环上存在Br或者I
- 寻找脱Cbz的一步反应，而环上卤素不脱掉

定义困难：

如何定义这个环系，杂环？C环？

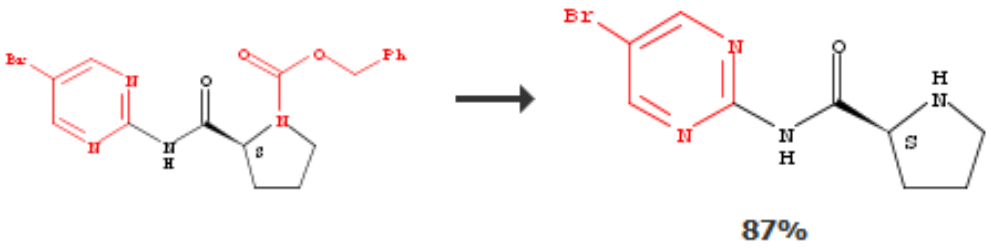
Cbz和环的连接方法

案例分析


Reaction Editor
Click bonds to be formed or broken during the reaction.
Atom
Short
reactant
product
reactant
Get reactions where the structure(s) are:
Variable only at the specified positions
Substructures of more complex structures
确定
取消
Scale 100
Formula not available

案例分析

32. **1 Hits in this Reference** **Similar Reactions**
Single Step *Hover over any structure for more options.*

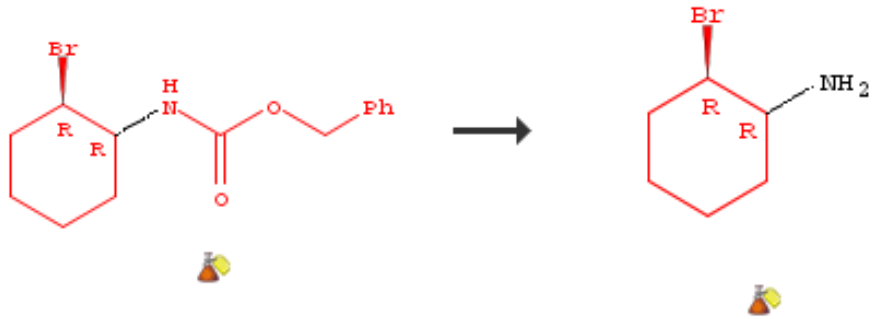


▸ Overview

▸ Experimental Procedure

存在我们想要的结果集

33. **1 Hits in this Reference** **Similar Reactions**
Single Step *Hover over any structure for more options.*

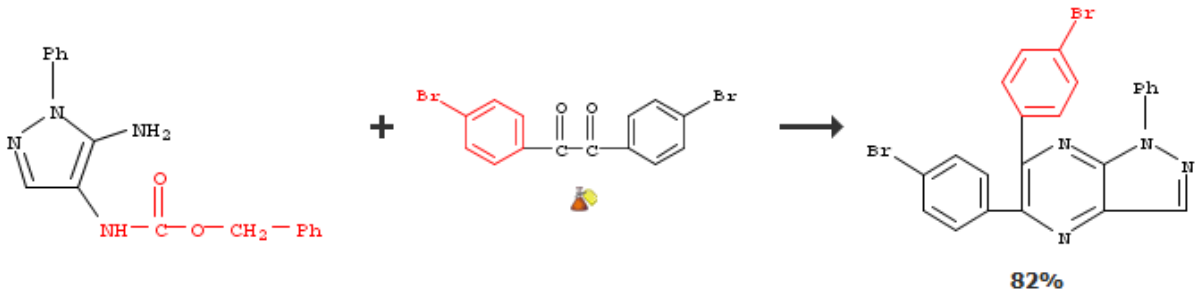


▸ Overview

▸ Experimental Procedure

案例分析

36. **1 Hits in this Reference** **Similar Reactions**
Single Step *Hover over any structure for more options.*



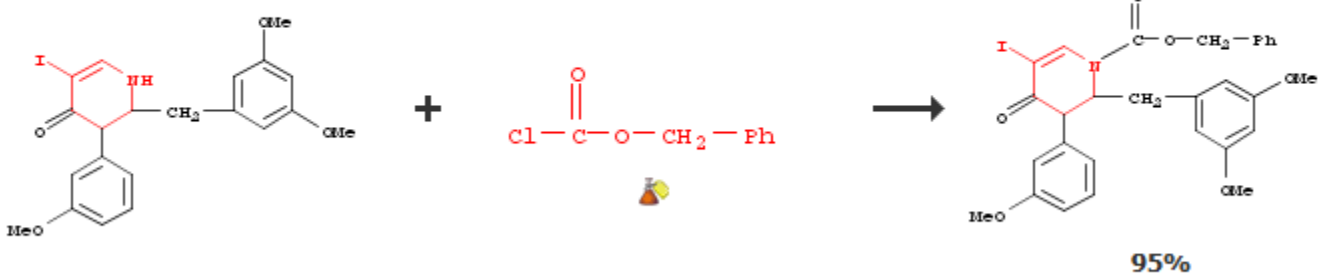
Overview

Experimental Procedure

存在大量的环与Cbz不在一个结构上的反应

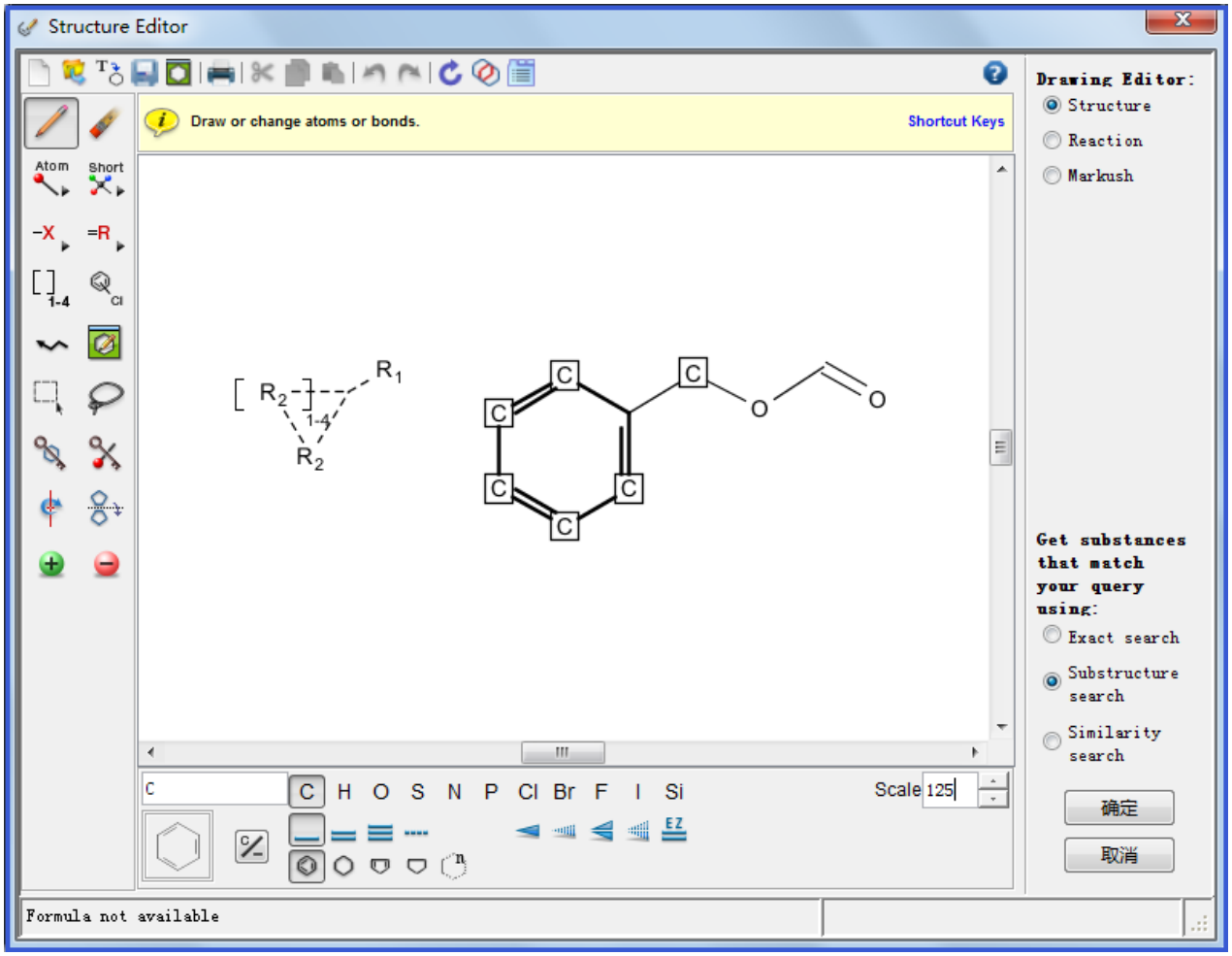
如何去除？

38. **1 Hits in this Reference** **Similar Reactions**
Single Step *Hover over any structure for more options.*



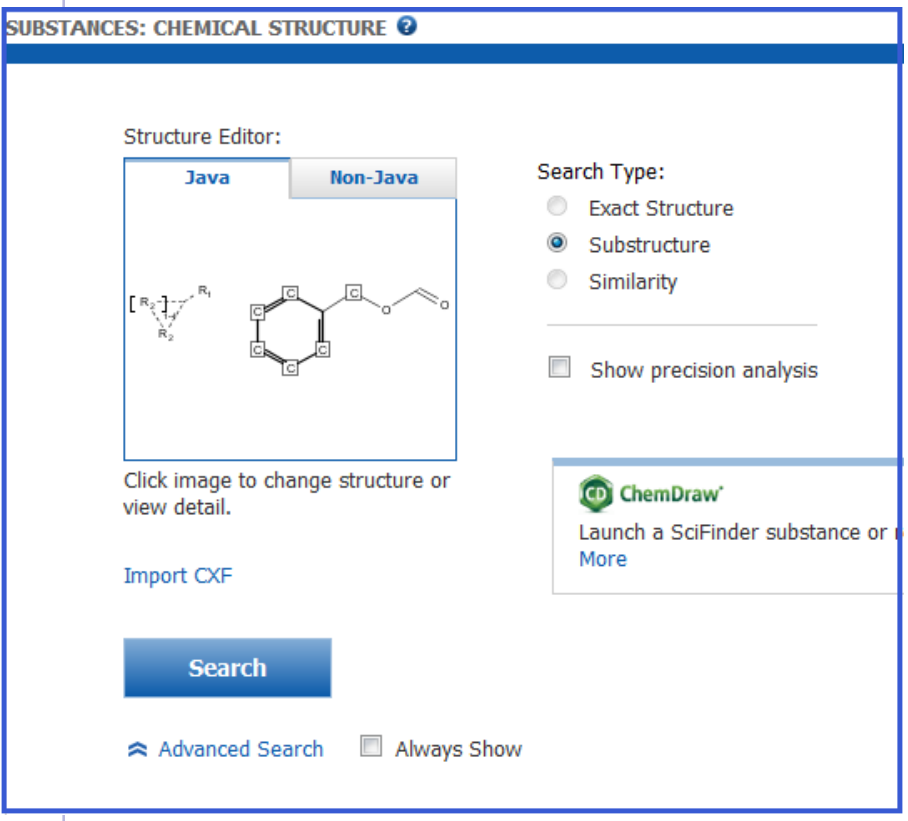
Overview

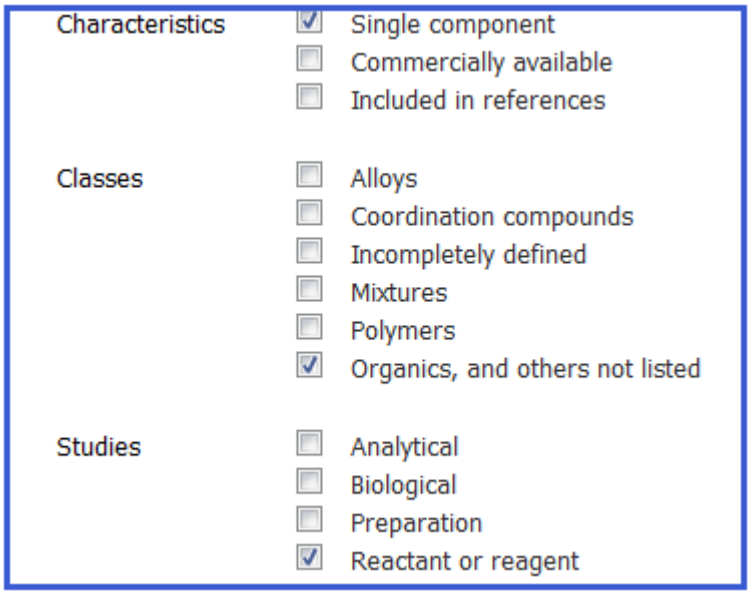
案例分析



这种类型的反应，需要先去检索和反应相关的物质

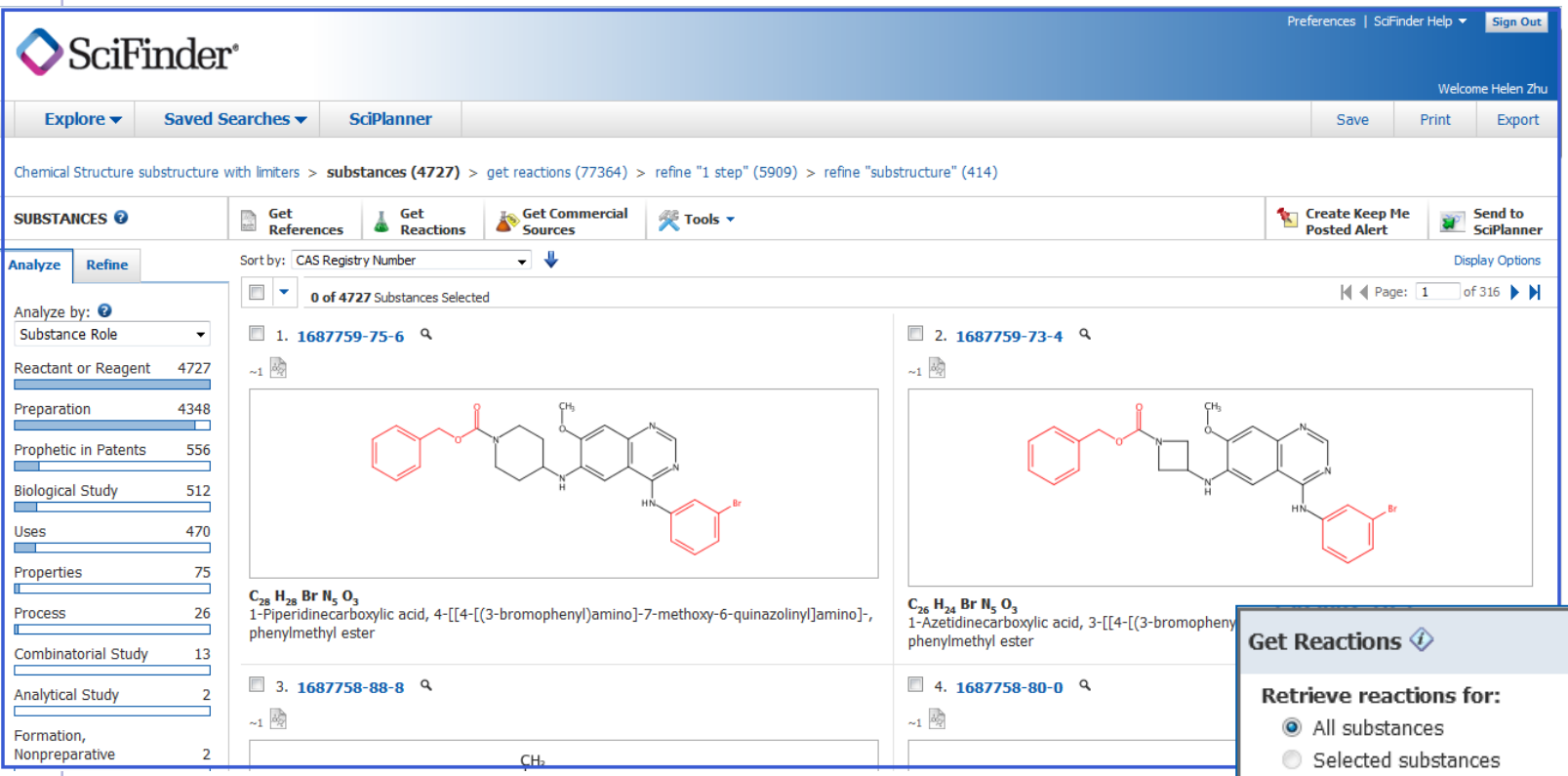
案例分析

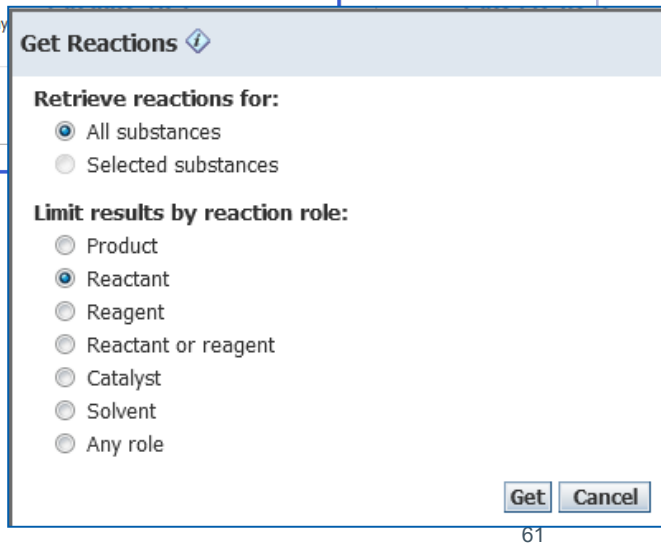




需要定义为单一组分和具备反应物或试剂角色的物质

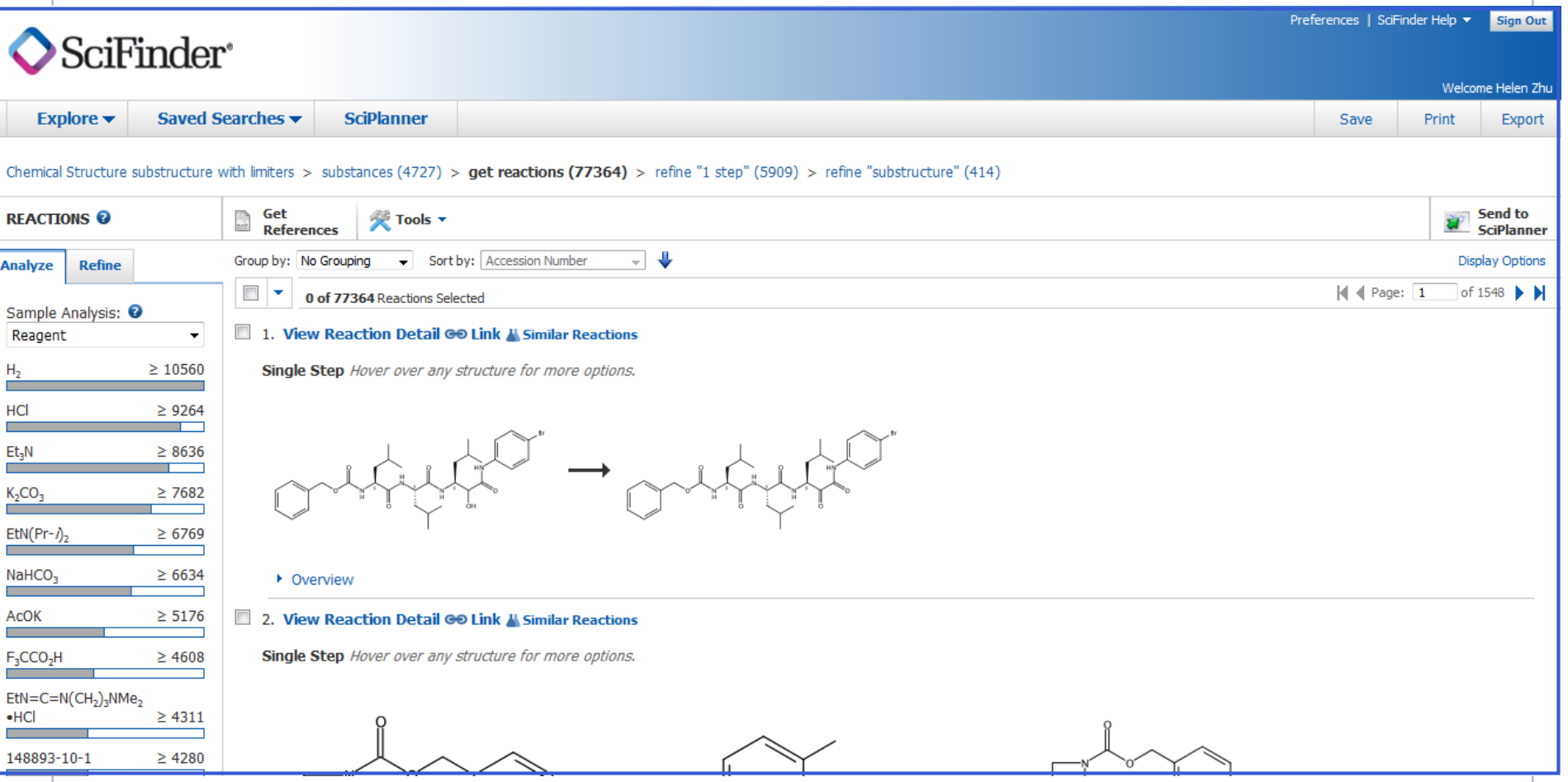
案例分析





获得所有可能的反应物，并获得这些物质作为反应物的反应

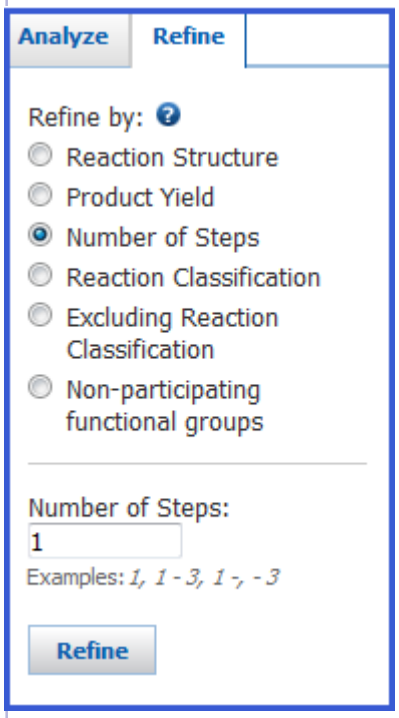
案例分析



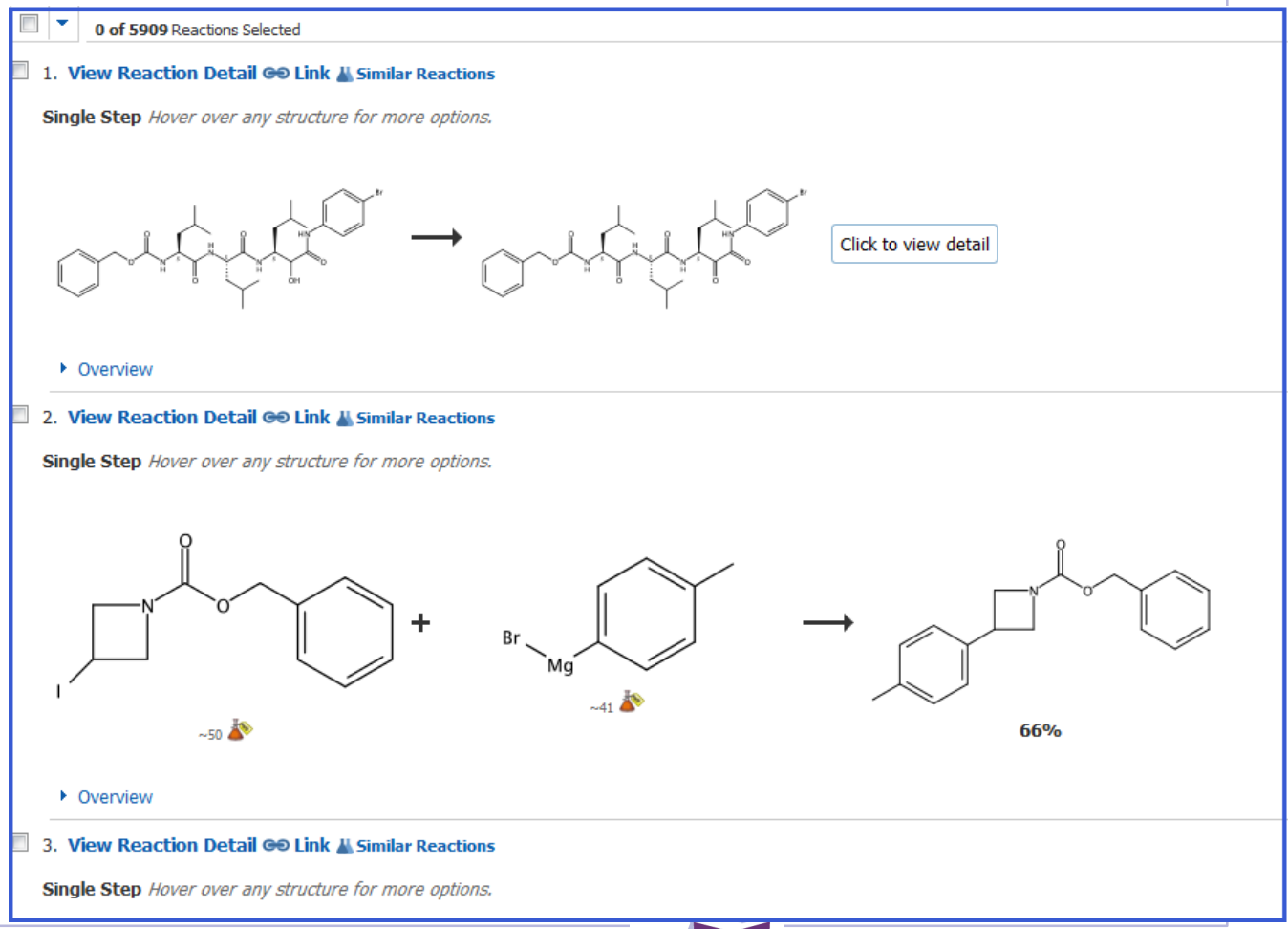
这些反应中的反应物都包含要求的结构，

需进一步把产物限定进去。

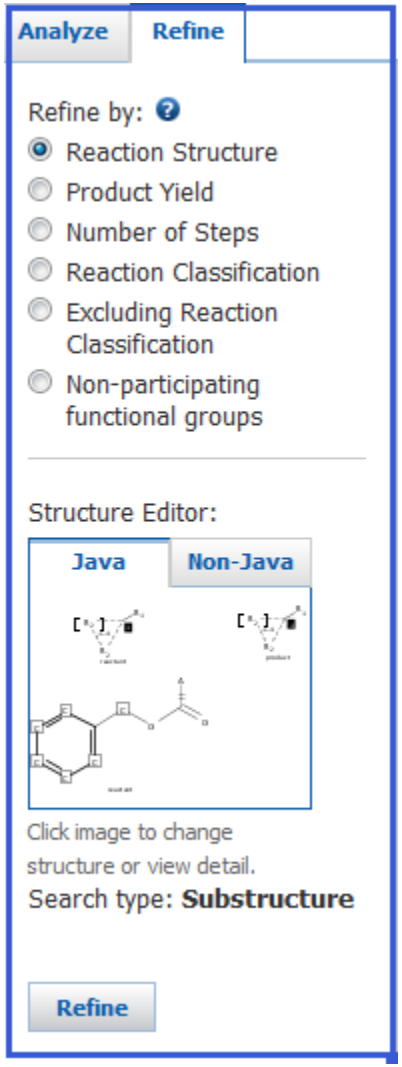
案例分析

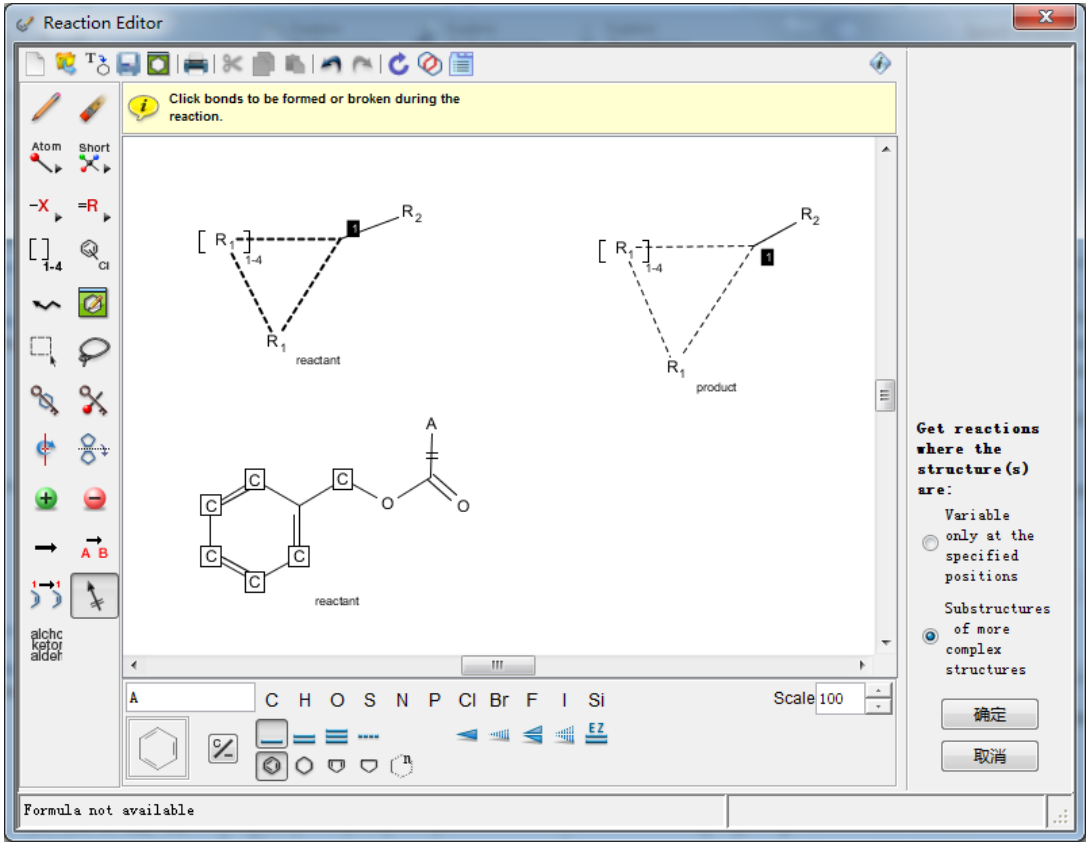


先限定反应步数为一步反应



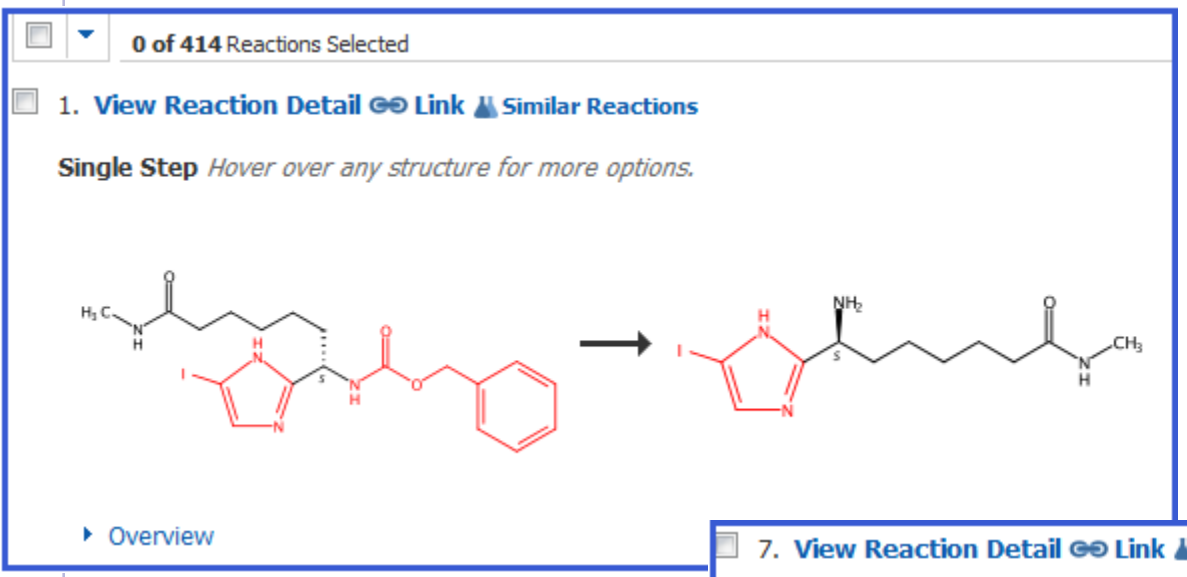
案例分析



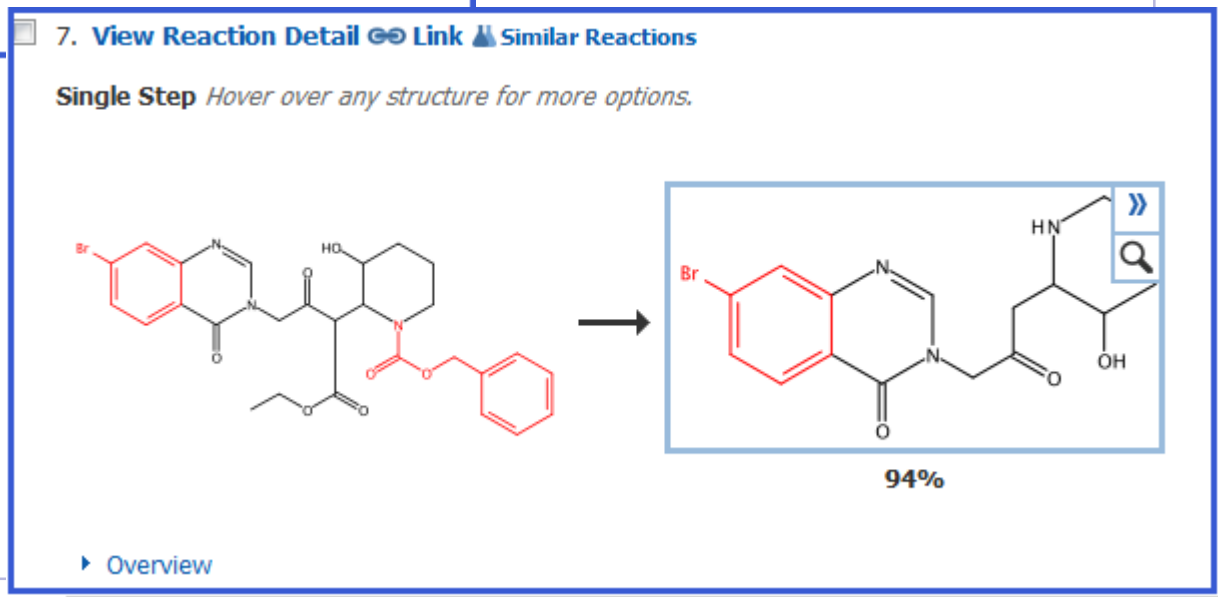


再做反应中心限定，沿用之前的定义

案例分析



都是我们想要的反应结果

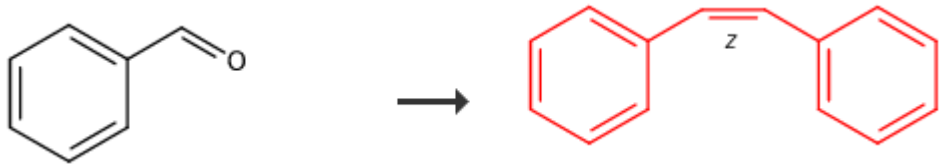


SciFinder中的检索思路

- 初步检索
- 浏览结果集，判断是否符合要求，利用系统工具限定结果
- 修正检索式，再次检索
- 浏览结果集，判断

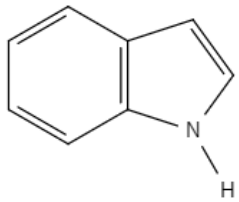
上机练习

1. 检索以下反应：



2. 底物上有伯羟基和仲羟基，能否找到合适的氧化剂使伯羟基氧化成醛而仲羟基不受影响？

3. 检索保护吲哚环上N原子而环上的羟基不受影响的反应？



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美国化学文摘社北京代表处

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