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Contents

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Chemistry Chat

Nine Short Stories - Part 3 -

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As you can see from the fact that Tokyo Chemical Industry Co., Ltd. sells more than 30,000 products, there are many different types of reagents. Naturally, the properties of the reagents vary widely. While some reagents can be handled without special care, others are vulnerable to moisture, oxygen, light, and heat and need to be handled with care. This time, I would like to talk about the storage of reagents.

Story 7. The veil of mystery

When Fumiko found the desired halogen compound in the reagent cabinet, she noticed the bottle was wrapped in black paper unlike the other reagent bottles. When she weighed the reagent on the balance, the black paper was in the way. Thus, Fumiko tore off the annoying paper. At this time, the reaction proceeded efficiently, but the yield of the product decreased with each reaction, and finally the reaction did not proceed at all.

⇨ Some of the reagents decompose when exposed to light. A brown bottle can often inhibit decomposition, but some of them are sensitive, and in such cases, they may be wrapped in black paper to protect them from light. Fumiko removed it because it was in the way of taking the reagent from the bottle. So, it is no wonder that **the reagent in the bottle was decomposing and the reaction was no longer proceeding**. Fumiko may think the bottle was shrouded in a veil of mystery, but the reagent manufacturers are not doing this to harass users, they are doing it for a good reason. If you are not sure, you should not act on your own judgment, but should consult with a professor.

Story 8. Time sense

Lab members were organizing the reagents stored in the laboratory. This included not only the reagent racks but also the reagents stored in a refrigerator. Mizuki was in charge of the refrigerator. He took out the reagents and put them on the lab bench and continued to check the labels with his colleague against the storage list. If the letters on the labels were hard to read, he would replace them with new ones. Mizuki picked up a bottle with an illegible label and tried to read the name of the reagent. At that moment, the lid blew off with a clunk and the reagent inside blew up vigorously, leaving a brown stain on the ceiling.

⇨ Some reagents are heat-sensitive and decompose. Such reagents are designated for refrigerated or frozen storage, so a refrigerator is necessary in laboratories. Mizuki considered that **they would be fine for a short period of time and put them on the lab bench, but it must have been too long, long enough for the reagents to decompose**. Since the reagent reached the ceiling, the inside of the bottle must have been pressurized to a very high level. If the bottle had been facing Mizuki, it might have brought about a horrible accident that I do not even want to imagine.

Story 9. Rusting atmosphere

Fumiko finished using acid chloride for her reaction, and returned the reagent bottle to the reagent rack. The next morning, when Fumiko looked in the reagent rack to take out the reagent, she found something brown, unlike yesterday. The metal rods to prevent the reagent bottles from tipping over were all rusty. Looking at the reagent bottle she used yesterday, she saw that the lid was a little loose and water drops were adhered to it. Fumiko understood it was obviously her own fault. She apologized to the other members and asked them to help her remove the rust of the metal rod.

⇨ There is an old haiku like "a razor rusts in one night on the rainy day in May". Indeed, rust can occur overnight if conditions are proper, such as humidity and temperature. In addition, the formation of rust is accelerated when the metal surface is exposed to acid corrosion. **If the lid of a bottle of acid chloride is loose, the moisture in the air will decompose it, producing hydrogen chloride, and the next day any metal surface may be covered in rust**. The lid of the reagent bottle must be tightened and further wrapped with tape. If space is available, it may be a good idea to store such reagents in a draft chamber. As much as possible, you do not want to make a mistake that would cause problems for others.

Author Information

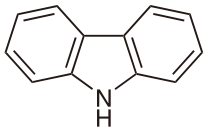
Professor Nagatoshi Nishiwaki received a Ph.D. in 1991 from Osaka University. He worked in Professor Ariga's group in the Department of Chemistry, Osaka Kyoiku University, as an assistant professor (1991-2000) and associate professor (2001-2008). From 2000 to 2001, he was with Karl Anker Jørgensen's group at Århus (Aarhus) University in Denmark. He worked at the Center for Collaborative Research, Anan National College of Technology, as an associate professor from 2008 to 2009. Then, he moved to the School of Environmental Science and Engineering, Kochi University of Technology in 2009, where he has been a professor since 2011. His research interests comprise synthetic organic chemistry using nitro compounds, heterocycles (synthesis, ring transformation, 1,3-dipolar cycloaddition, application as tools in organic synthesis), and pseudo-intramolecular reactions.

New Products Information

High Purity Carbazole with Excellent Optical Properties

Carbazole (high quality) (1)

Product Number: **C3722**
1g 5g 25g



(**1**)

Carbazole is an important building block in materials science. It has been recently reported that the optical properties of carbazole can be significantly affected by trace impurities that are difficult to separate.[1,2] The validity of the optical properties of carbazole derivatives containing impurities used as raw materials has also been discussed. In view of the importance of the purity of carbazole as a starting material, TCI has commercialized **1** with excellent optical properties. **1** is free from trace impurities that affect optical properties, and its optical properties are guaranteed. For example, the absorbance edge of **1** is flat. As shown in Figure 1, conventional carbazole shows the absorbance at 350 – 400 nm and room temperature phosphorescence (RTP) at 500 – 800 due to impurities. In contrast, **1** shows no RTP at that range.

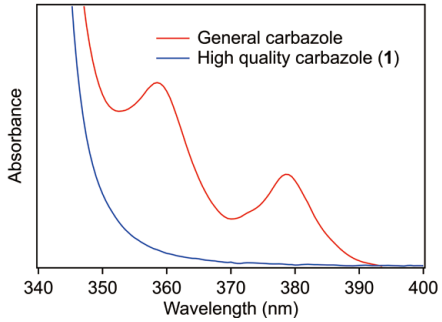


Figure 1. Comparison of absorption spectra of general carbazole and **1**

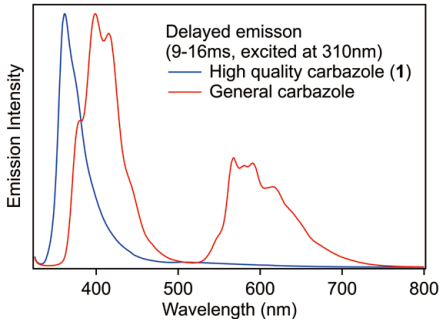


Figure 2. Comparison of emission spectra of general carbazole and **1**

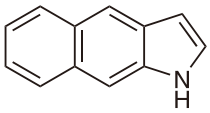
References

1) C. Chen, Z. Chi, K. C. Chong, A. S. Batsanov, Z. Yang, Z. Mao, Z. Yang, B. Liu, *Nat. Mater.* **2021**, *20*, 175.
2) C. Chen, K. C. Chong, Y. Pan, G. Qi, S. Xu, B. Liu, *ACS Mater. Lett.* **2021**, *3*, 1081.

Room Temperature Phosphorescence Inducer

1*H*-Benzo[*f*]indole (1)

Product Number: **B6283**
1g



(**1**)

1*H*-Benzo[*f*]indole (**1**) is a carbazole isomer found in trace amounts in carbazole from crude oil. Liu *et al*. reported that carbazole from crude oil, which contains **1**, shows room temperature phosphorescence (RTP). They also showed the expression of RTP by adding small amounts of **1** to **1**-free synthetic carbazole and confirmed **1** as a RTP inducer. Absorption and fluorescence properties of **1** are shown in Figure 1 and summarized in Table 1. On the other hand, as mentioned in the previous article and Figure 2, high quality carbazole (**2**) shows no RTP, but doping 0.5 wt% of **1** to **2** induces RTP at around 500 – 700 nm. **1** can be also used as a starting material for another RTP inducer.

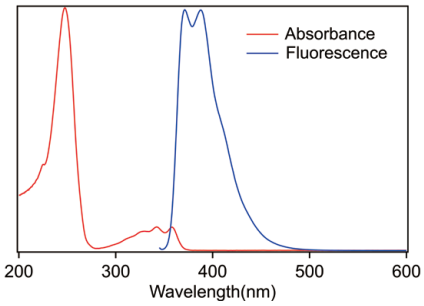


Figure 1. Absorption and fluorescence of **1**

	λ_{abs} / nm (ε / $M^{-1}cm^{-1}$)	λ_{em} / nm	Stokes shift (nm)
1	248 (64800), 330 (5058), 342 (6200), 358 (6100)	371, 388	13
2	236 (45200), 257 (16700), 292 (19200), 321 (4322), 334 (3743)	339, 354	5

Table 1. Comparison of optical properties of **1** with carbazole.

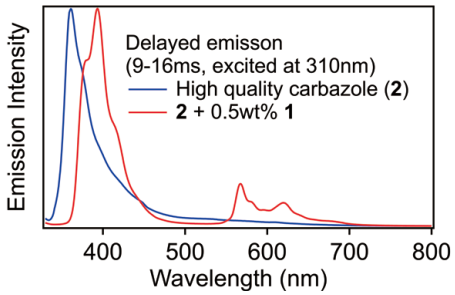


Figure 2. Difference of emission properties of **2** and **1**-doped **2**.

References

1) B. Liu, *Nat. Mater*. **2021**, *20*, 175.
2) C. Chen, K. C. Chong, Y. Pan, G. Qi, S. Xu, B. Liu, *ACS Mater. Lett*. **2021**, *3*, 1081.

Related Product

Carbazole (high quality) (**2**)	1g	5g	25g	C3722

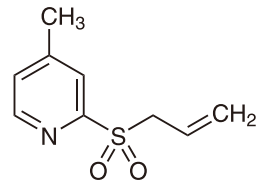
2-Allylsulfonyl Pyridines for Cross Coupling

2-(Allylsulfonyl)-4-methylpyridine (1) Product Number: **A3343**
200mg 1g

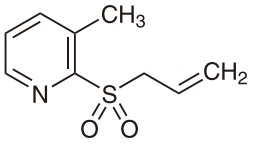
2-(Allylsulfonyl)-3-methylpyridine (2) Product Number: **A3344**
200mg 1g

2-(Allylsulfonyl)-5-methylpyridine (3) Product Number: **A3349**
200mg 1g

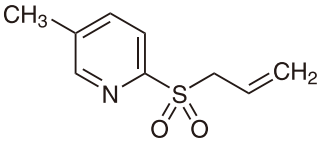
The use of pyridine derivatives in efficient C-C cross-coupling processes is useful in medicinal chemistry, and reactions using pyridine sulfinates have been reported.[1] Allylsulfonyl pyridines (**1**,**2**,**3**) are reagents improved by Willis *et al*.[2] Since these reagents function as potential pyridine sulfinate salts, they react with aryl halides to give 2-pyridylarenes by the palladium-catalyzed C-C cross-coupling reaction. It is also possible to convert another functional group of the pyridine ring because the allylsulfonyl group is more stable against general synthetic transformations than pyridine sulfinates.



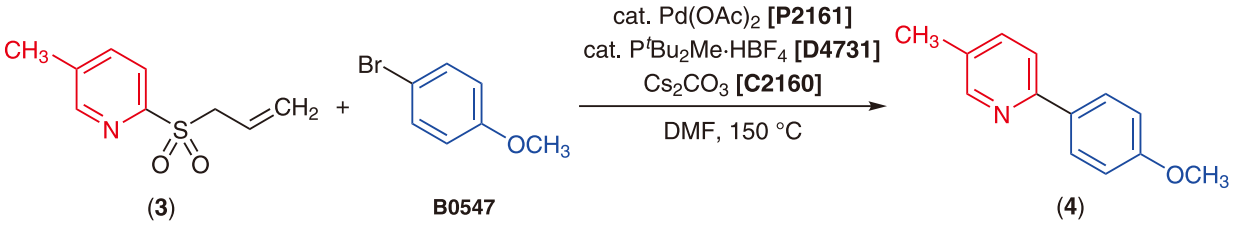
(**1**)



(**2**)



(**3**)



(**3**) **B0547** (**4**)

TCI Practical Example: Cross-coupling reaction using **3** as a nucleophile
To a pressure resistant test tube was charged with **3** (592 mg, 3.0 mmol, 1.5 equiv.), palladium(II) acetate (22 mg, 0.1 mmol, 5 mol%), $P^tBu_2Me{\cdot}HBF_4$ (50 mg, 0.2 mmol, 10 mol%), cesium carbonate (1.30 g, 4.0 mmol, 2.0 equiv.). The test tube was purged with nitrogen and DMF (10 mL) and 4-bromoanisole (0.25 mL, 2.0 mmol, 1.0 equiv.) were added in one portion at room temperature. The resulting mixture was stirred at 150 °C for 17 h. The reaction mixture was cooled to room temperature and quenched with water (10 mL). The organic layer was extracted with diethyl ether (100 mL x 3), dried with sodium sulfate (20 g) and then concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (ethyl acetate:hexane = 5:95 - 18:82) to afford the corresponding compound **4** as yellow solid (196 mg, 49%).

References

1) T. Markovic, B. N. Rocke, D. C. Blakemore, V. Mascitti, M. C. Willis, *Chem. Sci.* **2017**, *8*, 4437.
2) T. Markovic, P. R. D. Murray, B. N. Rocke, A. Shavnya, D. C. Blakemore, M. C. Willis, *J. Am. Chem. Soc.* **2018**, *140*, 15916.

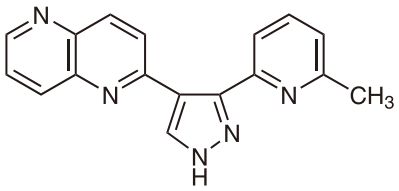
Related Products

Palladium(II) Acetate (Purified)			1g	P2161
Di-*tert*-butyl(methyl)phosphonium Tetrafluoroborate (= $P^tBu_2Me{\cdot}HBF_4$)		1g	5g	D4731
Cesium Carbonate		25	100g	C2160
4-Bromoanisole	25g	100g	500g	B0547

TGF-β Type I Receptor Inhibitor

RepSox (1)

Product Number: **R0224**
5mg 25mg



(**1**)

Table 1. Inhibition properties of **1** against ALK5[1)]

Assays	IC_{50} (nM)
ALK5 binding	23
ALK5 auto-phosphorylation	4
TGF-β cellular assay	18
p38 MAPK inhibition	>16000

RepSox (**1**) is a cell permeable TGF-β type I receptor inhibitor. **1** shows inhibition properties on ALK5 as shown in Table 1 and less potent activity against 9 other related kinases, including p38 MAPK, JNK1, and GSK3 (IC_{50} >16 μM).[1)] **1** promotes reprogramming of human and mouse embryonic fibroblasts to induced pluripotent stem cells (iPSCs).[2,3)] In addition, it was also reported that **1** induces human ESCs and iPSCs into glucagon-producing cells and insulin-producing cells, respectively.[4,5)]

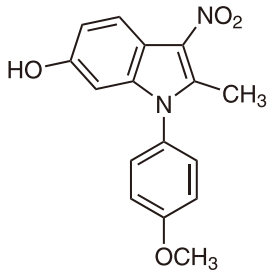
References

1) F. Gellibert, J. Woolven, M.-H. Fouchet, N. Mathews, H. Goodland, V. Lovegrove, A. Laroze, V.-L. Nguyen, S. Sautet, R. Wang, C. Janson, W. Smith, G. Krysa, V. Boullay, A.-C. de Gouville, S. Huet, D. Hartley, *J. Med. Chem.* **2004**, *47*, 4494.
2) J. K. Ichida, J. Blanchard, K. Lam, E. Y. Son, J. E. Chung, D. Egli, K. M. Loh, A. C. Carter, F. P. Di Giorgio, K. Koszka, D. Huangfu, H. Akutsu, D.R. Liu, L. L. Rubin, K. Eggan, *Cell Stem Cell* **2009**, *5*, 491.
3) D. Subramanyam, S. Lamouille, R. L. Judson, J. Y. Liu, N. Bucay, R. Derynck, R. Blelloch, *Nat. Biotechnol.* **2011**, *29*, 443.
4) Y. Kunisada, N, Tsubooka-Yamazoe, M. Shoji, M. Hosoya, *Stem Cell Res.* **2012**, *8*, 274.
5) A. Rezania, M. J. Riedel, R. D. Wideman, F. Karanu, Z. Ao, G. L. Warnock, T. J. Kieffer, *Diabetes* **2011**, *60*, 239.

Dual-Specificity Tyrosine Phosphorylation-Regulated Kinase (DYRK) Inhibitor

ID 8 (1)

Product Number: **I1108**
10mg 50mg



(**1**)

ID 8 (**1**) was reported as an indole derivative that can sustain embryonic stem cell self-renewal in long-term culture.[1)]

1 inhibits dual-specificity tyrosine phosphorylation-regulated kinase (DYRK).[2)] 1 supports Wnt-induced human embryonic stem cell proliferation and survival without fibroblast growth factor (FGF) and tumor growth factor-β (TGF-β) supplementation.[2)]

References

1) T. Miyabayashi, M. Yamamoto, A. Sato, S. Sakano, Y. Takahashi, *Biosci. Biotechnol. Biochem.* **2008**, *72*, 1242.
2) K. Hasegawa, S. Yasuda, J.-L. Teo, C. Nguyen, M. McMillan, C.-L. Hsieh, H. Suemori, N. Nakatsuji, M. Yamamoto, T. Miyabayashi, C. Lutzko, M. F. Pera, M. Kahn, *Stem Cells Transl. Med.* **2012**, *1*, 18.

Ready-to-Use Solution for Protein Extraction from *E. coli* / Yeast Cells

E.coli / Yeast Protein Extraction Buffer (1)

Product Number: **Y0021**
100mL

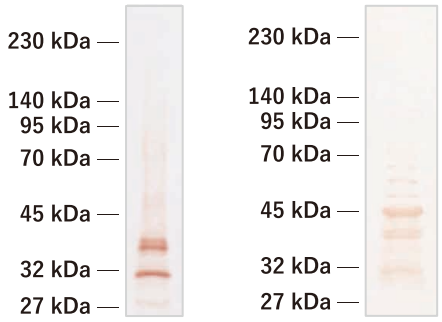
1 is a ready-to-use solution for protein extraction from cultured *Escherichia coli* (*E. coli*) / yeast cells. By suspending cells in **1** and then centrifuging, the supernatant containing proteins can be obtained. Extracted protein can be used in downstream applications such as electrophoresis and western blotting.

Application example
[Protein extraction]
1. Centrifuge *E. coli* at 5000 × g and yeast at 3000 × g for 10 minutes.
2. Remove as much medium as possible from the pellet.
3. Resuspend pellet in **1**. Use 2-4 mL buffer per gram of pellet and mix at room temperature for 10 minutes.
4. Centrifuge *E. coli* at 15000 × g and yeast at 14000 × g for 10 minutes and collect lysate fraction.

[Protein quantification]: measurement of protein concentration in lysate fraction by pyrogallol red method

	When using **1**	When using PBS
E. coli K-12	3.90 mg/mL	0 mg/mL
S. cerevisiae Y-12632	2.79 mg/mL	0 mg/mL

[Electrophoresis / Western blotting]: Detection of specific protein in lysate fraction



RecA in *E. coli* (left, 38 kDa) and Rad51 in *S. cerevisiae* (right, 43 kDa) can be extracted efficiently.

References

1) L. Vuillard, C. Braun-Breton, T. Rabilloud, *Biochem. J.* **1995**, *305*, 337.
2) V. N. Danilevich, L. E. Petrovskaya, E. V. Grishin, *Chem. Eng. Technol.* **2008**, *31*, 904.

Related Products

Pyrogallol Red (Ready-to-use solution) [for Protein determination]	100mL	P2575
DAB staining kit	1KIT	D5909

Ready-to-use Solution for Protein Extraction from Nervous Tissue

Nervous Tissue Protein Extraction Buffer (1)

Product Number: **B6279**
100mL

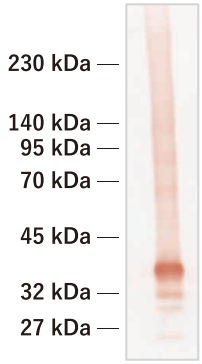
1 is a ready-to-use solution for protein extraction from nervous tissue. By suspending tissue in **1** and then centrifuging, the supernatant containing proteins can be obtained. Extracted protein can be used in downstream applications such as electrophoresis and western blotting.

Application example
[Protein extraction]
1. Wash mouse brain twice with PBS.
2. Weigh samples. For each gram of sample, add 10 mL of **1** and homogenize.
3. Incubate on ice for 10 minutes.
4. Centrifuge the sample at 10000 × g for 10 minutes at 4 °C and collect lysate fraction.

[Protein quantification]: measurement of protein concentration in lysate fraction by pyrogallol red method

	When using **1**	When using PBS
Mouse brain	3.63 mg/mL	1.59 mg/mL

[Electrophoresis / Western blotting]: Detection of specific protein in lysate fraction



Synaptophysin (38 kDa) in synaptic vesicle can be extracted from mouse brain efficiently.

References

1) S. Musunuri, M. Wetterhall, M. Ingelsson, L. Lannfelt, K. Artemenko, J. Bergquist, K. Kultima, G. Shevchenko, *J. Proteome Res.* **2014**, *13*, 2056.
2) C. Ericsson, I. Peredo, M. Nistér, *Acta Oncol.* **2007**, *46*, 10.

Related Products

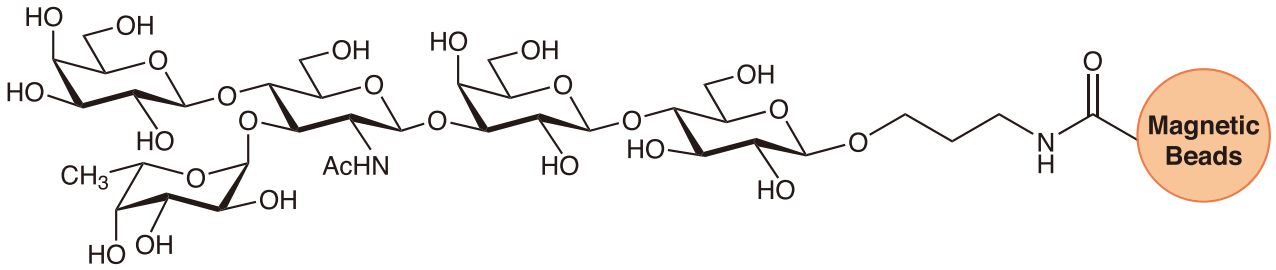
Pyrogallol Red (Ready-to-use solution) [for Protein determination]	100mL	P2575
DAB staining kit	1KIT	D5909

Carbohydrate-Conjugated Magnetic Beads

Lewis X-Magnetic Beads (1)

Product Number: **L0381**
1VIAL

Carbohydrate-conjugated magnetic beads are a highly useful tool for isolation of cells and glycan-binding molecules[1,2] and **1** are superparamagnetic beads conjugated to stringently purified synthetic glycan. **1** have an average particle size of 1 μm, and can be used in a variety of applications like flow cytometry. In addition, the glycan moiety is bonded to the magnetic beads by a unique conjugation method, which does not require ring opening at the reducing end of the oligosaccharide. Therefore, **1** maintain a native carbohydrate structure. In addition to the glycan conjugates introduced here, custom synthesis of magnetic beads can be applied to meet customers' requests.



(**1**)

References

1) X.-L. Sun, W. Cui, C. Haller, E. L. Chaikof, *ChemBioChem* **2004**, *5*, 1593.
2) P. D. Rye, N. V. Bovin, *Glycobiology* **1997**, *7*, 179.

Related Products

Anti-Lewis X Monoclonal Antibody	1VIAL	A2578
HSA-Lewis X	1VIAL	H1719

Recombinant Antiviral Lectin GRFT

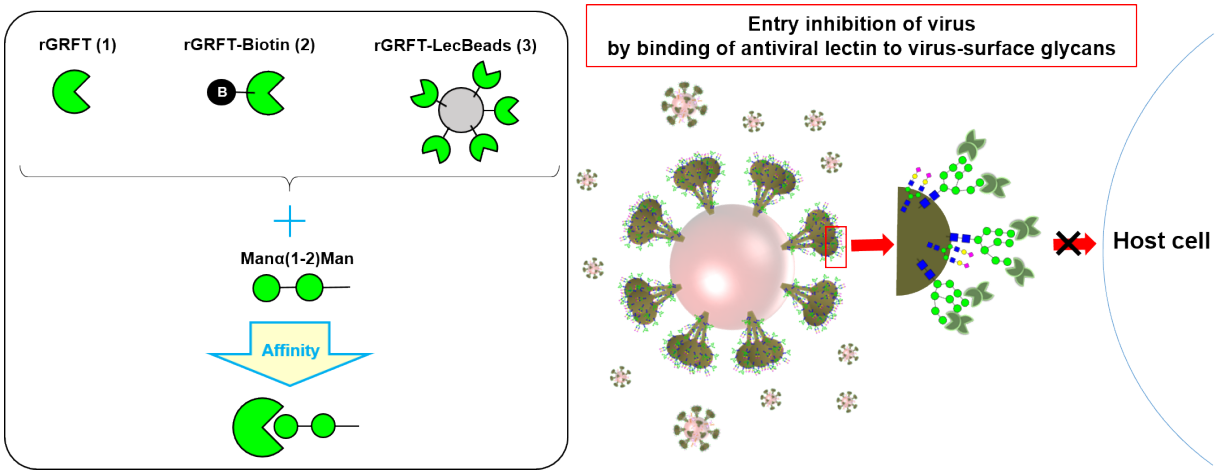
Recombinant *Griffithsia* sp. lectin (= rGRFT) (1) Product Number: **R0229** **1mL**

rGRFT-Biotin [for Manα(1-2)Man] (2) Product Number: **R0234** **1mL**

rGRFT-LecBeads [for Manα(1-2)Man] (3) Product Number: **R0239** **1VIAL**

The GRFT (**1**) known as Griffithsin is an α-mannose-binding lectin derived from red algae. Its antiviral activity against various viruses such as HIV, SARS-CoV, HCV, and Ebola virus, has been reported.[1,2] The antiviral function of lectins is generally thought to interfere with the entry of virus particles into cells through specific binding to oligosaccharide structures of glycoproteins on the virus surface, and an interaction of GRFT with SARS-CoV-2 has also been suggested.[3]

In addition to an *Escherichia coli* recombinant GRFT, we have a lineup of biotinylated rGRFT-Biotin (**2**), which is effective for detection and immunoprecipitation, and rGRFT-LecBeads (**3**), which are immobilized on agarose for capturing glycoproteins such as viruses.



rGRFT were commercialized under license from National Institute of Advanced Industrial Science and Technology (AIST). rGRFT-LecBeads were developed in collaboration with AIST.

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DOI: https://doi.org/10.1080/07391102.2020.1851303

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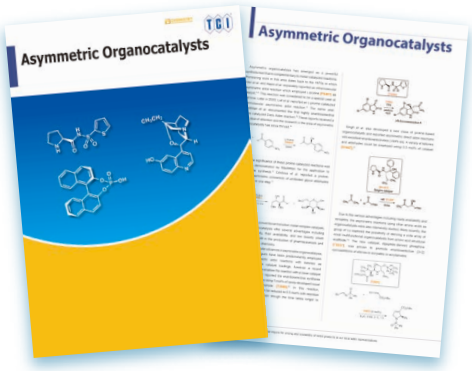
Pamphlets of TCI Products



Asymmetric Organocatalysts

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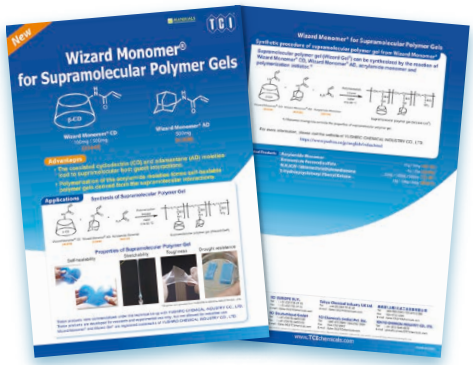

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