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Palladium-loaded renewable polymer as a green heterogeneous catalyst for cross-coupling reactions under microwave irradiation

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A new palladium catalyst was prepared by immobilising ligand 2, 2'-dipyridylamine on the backbone of an acidic rosin polymer from gum rosin, on to which palladium(II) was bound *via* coordination. The catalyst at a low loading of 0.2 mol% was found to be highly effective for Suzuki–Miyaura coupling reactions of aryl halides and arylboronic acids under microwave irradiation in the presence of 1 equiv. of Na_2CO_3, affording excellent yields of the corresponding biaryls. Moreover, the catalyst exhibited very good recyclability over three cycles.

Keywords: renewable polymer, palladium, heterogeneous catalysts, Suzuki–Miyaura reaction, microwave irradiation

In the past 30 years, palladium-mediated cross-coupling reactions have become popular as convenient techniques for the formation of carbon–carbon bonds.[1–6] There have been several reports of highly-active catalysts that can be used in various homogeneous C–C cross-coupling reactions (see refs 7–10). Despite the considerable progress in homogeneous catalysis that has been made, problems relating to the separation and recycling of the catalyst persist. A promising approach for achieving this goal is the development of heterogeneous catalytic systems giving scope for efficient recovery and repeated use in green processes.[11–15] In this context several studies have reported the synthesis of heterogeneous palladium catalysts, *e.g.*, based on palladium-loaded polymers,[16] palladium-loaded inorganic solids,[17,18] and dendrimers.[19,20] In addition, renewable polymers have become a rapidly growing area, as these materials could potentially replace or partially replace environmentally and energy unfavourable petroleum-derived polymers.[21–23]

We recently developed a new class of renewable polymers using gum rosin,[24–26] a natural resource abundantly available. We now report the use of a palladium-on-rosin catalyst in the Suzuki–Miyaura cross-coupling reaction of aryl halides and phenylboronic acids under microwave irradiation.

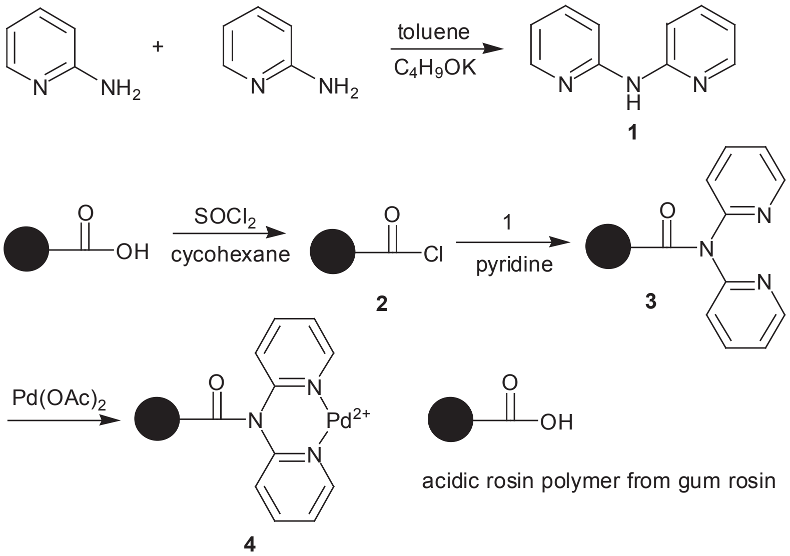
Results and discussion

Synthesis of the Pd-loaded polymer (Scheme 1)

2,2′-Dipyridylamine **1** was prepared by base-catalysed nucleophilic substitution of the chloro group of 2-chloropyridine by 2-pyridylamine. The renewable resin polymer was treated with thionyl chloride at 80 °C to convert the carboxylic acid residues to acid chlorides to form derivatised polymer **2**. Then **2** was treated with excess 2,2′-dipyridylamine and pyridine at 100 °C for 3 h. (Ligand 2,2′-dipyridylamine is known to possess a remarkable binding ability to transition metal ions.[27]) The product of this reaction was a 2,2′-dipyridylamidated polymer **3**. Finally, treatment of **3** with $Pd(OAc)_2$ yielded the palladium(II)-loaded polymer **4**.

Characterisation of the amidated polymer **3** *and Pd-loaded catalyst* **4**

The chemical conversion from renewable polymer to immobiled 2,2′-dipyridylamine renewable resin polymer **3** was verified by FTIR analysis as shown in Fig. 1. In the spectrum of the renewable polymer (a), the peaks attributed to the O–H bond of the carboxyl group in the polymer display around 3495 cm^{-1} and the C=O bond shows an absorption at 1743 cm^{-1}. The curve (b) of immobilised 2,2′-dipyridylamine on renewable resin polymer **3** shows the absence of the hydroxy band at 3495 cm^{-1}



Scheme 1

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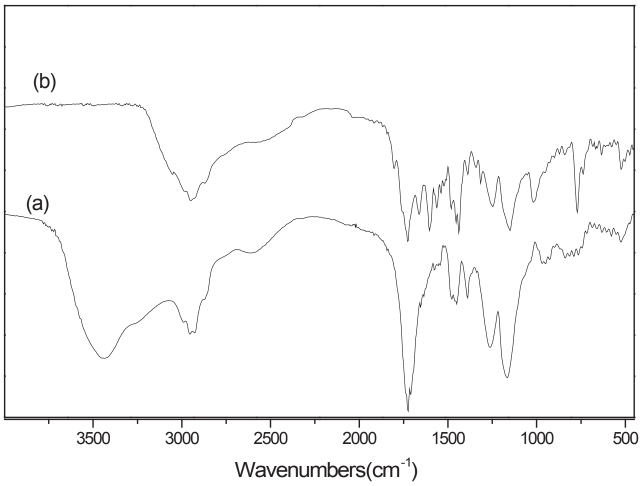


Fig. 1 IR spectra of (a) the renewable polymer and (b) the amidated polymer **3**.

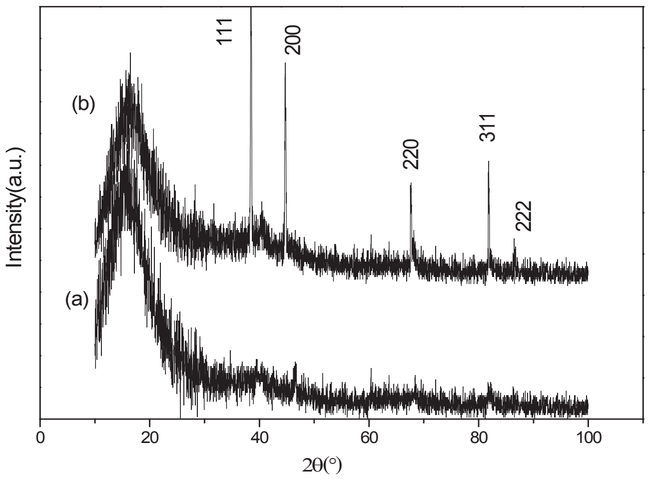


Fig. 2 Wide angle powder XRD image of (a) polymer **3** and (b) Pd-loaded catalyst **4**.

and the appearance of bands at 1662, 1604, 1563 cm^{-1} (amide, pyridine) which revealed that the amidation reaction had been successful.

The resulting Pd-loaded catalyst **4** was characterised by its XRD pattern (Fig. 2). The Bragg reflections at 2θ values of 39.9°, 46.5°, 68.3°, 82.3° and 86.6° correspond to the index face-centred cubic crystalline structure of the palladium (111), (200), (220), (311) and (222) lattice planes respectively, suggesting the presence of crystalline palladium.

The amount of immobilised Pd on the renewable rosin polymer **3** was quantified by ICP-AES. The loading level of Pd on the resin was found to be 0.093 mmol g^{-1}.

Since Suzuki–Miyaura reactions are usually carried out at high temperature, the thermal stability and recyclability of a catalyst is important. We therefore carried out a thermogravimetric (TG) analysis. The TG curves in Fig. 3 of polymer **3** and Pd-loaded catalyst **4** show that the catalyst systems were stable up to 225 °C under a nitrogen atmosphere. The first mass decrease was due to the release of assimilated water. This was followed by weight loss caused by oxidative decomposition of the rosin polymer.

Catalysis of the Suzuki–Miyaura reactions

Compared to traditional heating, microwave-heating can enhance the rate of reaction, reduce side reactions and increase product yields,[28–30] so we chose to employ this method to accelerate our reaction. Initially, the reaction conditions of the Suzuki–Miyaura reaction using the Pd-loaded catalyst **4** were optimised as regards the solvent and base using as a model the reaction between 4-bromoacetophenone (**5** X=Br, R^1=4-MeCO) and 4-methoxyphenylboronic acid (**6** R^2=MeO) in ethanol/water (1 : 1) as solvent (Scheme 2). Na_2CO_3 gave the highest yield of 4-methoxy-4′-acetylbiphenyl among the several bases tested, as Table 1 shows. Other inorganic bases, such as K_3PO_4, KOH and K_2CO_3 are slightly less efficient, while Et_3N gave the product in a much lower yield (entry 5). Moreover the reactions would not proceed without base (entry 6). From the solvent screening, (1 : 1) EtOH/H_2O was found to be optimal (entries 7–12). If just water was used, only moderate results were obtained (entry 12). We found that the solvent system is an important factor for the Suzuki–Miyaura in the heterogeneous catalytic system, as such a reaction requires both the compatibility of the solid support and the solubility of the organic and inorganic reagents.

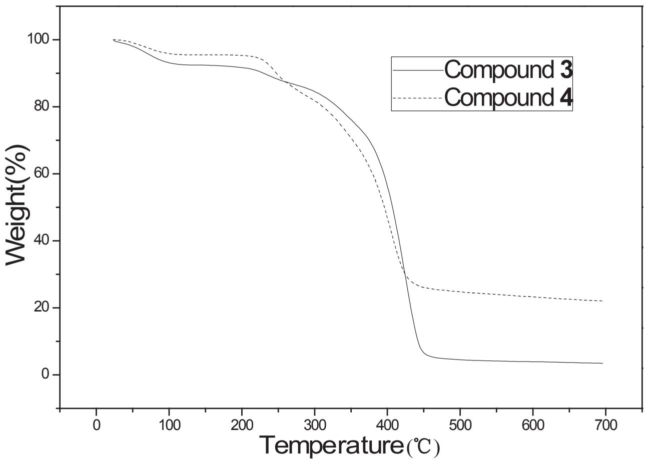
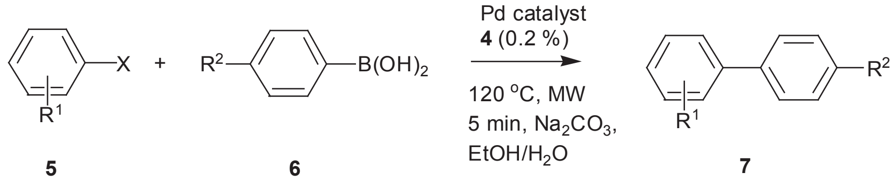


Fig. 3 Thermogravimetric curves of amidated polymer **3** and Pd-loaded catalyst **4**.

To evaluate the effectiveness of the microwave-assisted reaction, we carried out a comparison using the optimal conditions, between the catalysed coupling reaction of 4-bromo acetophenone with 4-methoxyphenylboronic acid, heated by conventional methods and microwave radiation. The results are listed in Table 2. It is clear that the microwave-assisted Suzuki–Miyaura reactions superior to conventional heating being faster and giving a greater yield.



Scheme 2

Table 1 The effect of varying the base and solvent in the Suzuki–Miyaura cross-coupling reaction of 4-bromoacetophenone (**5** X=Br, R^1=4-MeCO) with 4-methoxyphenylboronic acid (**6** R^2=MeO) under microwave irradiation (Scheme 2)[a]

Entry	Base	Solvent (1 : 1)	Yield/%[b]
1	Na_2CO_3	EtOH/H_2O	99.7
2	K_3PO_4	EtOH/H_2O	98.5
3	KOH	EtOH/H_2O	92.4
4	K_2CO_3	EtOH/H_2O	94.7
5	Et_3N	EtOH/H_2O	76.9
6	(without base)	EtOH/H_2O	0
7	Na_2CO_3	EtOH/H_2O	99.7
8	Na_2CO_3	DMF/H_2O	99.0
9	Na_2CO_3	DMAC/H_2O	98.0
10	Na_2CO_3	MeCN/H_2O	57.8
11	Na_2CO_3	MeCOMe/H_2O	95.7
12	Na_2CO_3	H_2O	77.2

[a] Reaction conditions:4-bromoacetophenone (**5** X=Br, R^1=4-MeCO) (0.5 mmol), 4-methoxy phenylboronic acid (**6** R^2=MeO) (0.65 mmol), base (1 mmol), solvent (2 mL) and 0.2 mol% Pd catalyst **4** were subjected to microwave irradiation (60 W) for 5 min at 120 °C. DMAC, *N,N*-Dimethylacetamide.

[b] Determined by HPLC analysis.

Table 2 The effect of varying the heating mode for Suzuki–Miyaura cross-coupling reaction of 4-bromoacetophenone (**5** X=Br, R^1=4-MeCO) with 4-methoxyphenylboronic acid (**6** R^2=MeO) (Scheme 2)[a]

Entry	Heating mode	Time/min	Yield/%[b]
1	Microwave irradiation	5	99.7
2	Conventional reflux	30	38.7
3	Conventional reflux	60	69.5
4	Conventional reflux	120	93.1

[a] Reaction conditions: 4-bromoacetophenone (0.5 mmol), 4-methoxy phenylboronic acid (0.65 mmol), Na_2CO_3 (1 mmol), EtOH/H_2O (1 : 1) (2 mL) and 0.2 mol% Pd catalyst **4** were subjected to heating at various temperatures or to microwave irradiation (60 W) for 5 min at 120 °C.

[b] Determined by HPLC analysis.

The catalytic system was applicable to a wide range of aryl halides and two different types of boronic acid (Scheme 2) (Table 3). The results show that the couplings of aryl bromides with arylboronic acids whether bearing an electron-donating or an electron-withdrawing group proceeded in good yield (entries 2–10). Similar results were obtained in the reaction of iodobenzene with arylboronic acids (entry 1). We also explored the scope of this reaction using aryl chlorides, and found that the conversion of aryl chloride was very good (87%, entry 11). The study of the coupling reaction using 4-methoxyphenylboronic was also carried out, and the corresponding products were produced in excellent yields (entries 13–24).

Catalyst recycling

To evaluate the reusability of the Pd-loaded catalyst **4**, a recycling test was carried out using the recovered catalysts from the Suzuki–Miyaura cross-coupling reaction of iodobenzene with 4-methoxyphenylboronic acid (Table 4). The reaction was carried out with Na_2CO_3 in a mixture of (1 : 1) EtOH/H_2O under microwave irradiation for 5 min. Upon completion of the reaction, products were extracted with dichloromethane and the yields were determined by HPLC. The resulting Pd catalyst **4** was reused with the same substrates for the next cycle. As can be seen from Table 4, the Pd-loaded catalyst **4** could be used three times with no significant decrease in the yields. But on the sixth cycle, the yield had dropped to 53%.

Conclusion

In conclusion, we have prepared a palladium catalyst loaded onto a renewable rosin polymer and applied it successfully to the Suzuki–Miyaura cross-coupling reactions of aryl halides and phenylboronic acids under microwave irradiation.

Table 3 Suzuki–Miyaura couplings of various aryl halides (5 X=Cl, Br or I; R^1=various) with phenyl-(6 R^2=H) or 4-methoxyphenyl-boronic acid (6 R^2=MeO) under the optimised conditions (Scheme 2)[a]

Entry	X	R^1	6	Product	Yield/%[b]
1	I	H	H	**7a**	98
2	Br	4-MeCO	H	**7b**	98
3	Br	4-CHO	H	**7c**	97
4	Br	3-CHO	H	**7d**	97
5	Br	2-CHO	H	**7e**	92
6	Br	4-OMe	H	**7f**	97
7	Br	4-CH_3	H	**7g**	95
8	Br	4-OH	H	**7h**	96
9	Br	4-Cl	H	**7i**	94
10	Br	H	H	**7j**	95
11	Cl	H	H	**7k**	87
12	Cl	4-NO_2	H	**7l**	71
13	I	H	OMe	**7m**	99
14	Br	4-MeCO	OMe	**7n**	98
15	Br	4-CHO	OMe	**7o**	97
16	Br	3-CHO	OMe	**7p**	98
17	Br	2-CHO	OMe	**7q**	96
18	Br	4-OMe	OMe	**7r**	97
19	Br	4-CH_3	OMe	**7s**	95
20	Br	4-OH	OMe	**7t**	96
21	Br	4-Cl	OMe	**7u**	95
22	Br	H	OMe	**7v**	98
23	Cl	H	OMe	**7w**	80
24	Cl	4-NO_2	OMe	**7x**	66

[a] Reaction conditions: aryl halide (0.5 mmol), phenyl-(**6** R^2=H) or 4-methoxyphenylboronic acid (**6** R^2=MeO) (0.65 mmol), Na_2CO_3 (1 mmol), EtOH/H_2O (1 : 1) (2 mL) and 0.2 mol% Pd-loaded catalyst **4** were subjected to microwave irradiation (60 W) for 5 min at 120 °C.

[b] Isolated yield after purification by column chromatography.

Table 4 Recycling efficiency of the Suzuki–Miyaura cross-coupling of iodobenzene (5 X=I; R^1=H) with 4-methoxyphenylboronic acid (**6** R^2=4-MeO) under the optimised conditions over six cycles (Scheme 2)[a]

Cycles/*n*	1	2	3	4	5	6
Conversion/%[b]	99.9	97	92.7	82.2	75.5	53

[a] Reaction conditions: iodobenzene (**5** X=I; R^1=H) (0.5 mmol), 4-methoxyphenylboronic acid (**6** R^2=MeO) (0.65 mmol), Na_2CO_3 (1 mmol), (EtOH/H_2O (1 : 1) (2 mL) and recycled 0.2 mol% Pd catalyst **4** were subjected to microwave irradiation (60 W) for 5 min at 120 °C.

[b] Determined by HPLC analysis.

Experimental

Unless otherwise noted, reagents and materials were commercially available and were used as received without further purification. Solvents were purified according to standard methods. The acidic rosin polymer (acid value: 4.464 mmol g^{-1}) was prepared according to established procedures.[26] Melting points were measured using a WRS-1B apparatus. The ^1H NMR spectra were recorded on a Bruker Avance 600 MHz spectrometer in $CDCl_3$ using TMS as internal standard. Chemical shifts (δ) are given in ppm and coupling constants (*J*) in Hz. The IR spectra were performed in the range 400–4000 cm^{-1} using KBr pellets on a Nicolet IS10 FTIR spectrometer. HPLC detection was carried out on a C18 reversed-phase column (5 µ, 200 × 4.6 mm) using MeOH : water (80 : 20)(v/v) as the mobile phase at a flow rate of 0.8 mL min^{-1}. Microwave reactions were carried out in a Biotage Initiator 60. X-ray powder diffraction (XRD) images were determined on a XD-3 and TGA analyses on a STA 449 F3. ICP-AES spectra data were recorded on an iCAP-6000 SERIES.

Synthesis of 2,2′-dipyridylamine **1**

Potassium *t*-butoxide (15.50 g, 138.1 mmol) was added to a solution of 2-aminopyridine (10.00 g, 106.2 mmol) and 2-chloropyridine

(13.27 g, 116.8 mmol) in toluene (100 mL), and the mixture was stirred at 120 °C for 36 h under a nitrogen atmosphere. After cooling to room temperature and removing solvent under vacuum, the crude product was extracted with ether and the combined ethers then washed with water three times. The organic layer was dried with magnesium sulfate, filtered and the solvent was removed under reduced pressure. The residue was purified by chromatography on silica gel to yield a white solid,[31] 13.73 g (75.5%). m.p. 90.3–91.6 °C (lit.[32] 94–95 °C), ^{1}H NMR: δ 8.28–8.27 (m, 2H), 8.19 (brs, 1H), 7.61–7.55 (m, 4H), 6.85–6.83 (m, 2H).

Synthesis of the amidated polymer **3**

After the rosin polymer (2.00 g, acid value: 4.46 mmol g^{-1}) was swelled in dry cyclohexane (20 mL) at room temperature for 4 h, thionyl chloride (3.19 g, 26.78 mmol) was added with cooling. Then the mixture was stirred at 80 °C for 3 h. After cooling to room temperature and removing the excess thionyl chloride, the residue was washed twice with cyclohexane to afford a white solid compound **2**. Then, pyridine (1.06 g, 13.4 mmol) was added to a mixture of compound **2** and 2,2′-dipyridylamine **1** (1.83 g, 10.7 mmol) in dry 1,4-dioxane (20 mL), and the reaction mixture was refluxed at 100 °C for 3 h. After removing solvent under reduced pressure, the residue was washed three times with water, and then dried in vacuum. A white solid compound **3** was obtained, yield 2.21 g; IR (KBr): 2949, 1726, 1662, 1604, 1563, 1438, 1247, 1150, 1018, 771 cm^{-1}.

Synthesis of Pd-loaded catalyst **4**

A mixture of amidated rosin polymer **3** (0.5 g) and palladium acetate (80 mg, 0.36 mmol) in methanol/water (1 : 1) (10 mL) was treated with concentrated hydrochloric acid (1 mL). Then a solution of sodium hydroxide (20%) was added slowly to adjust the pH to 5. The mixture was stirred at room temperature for 24 h. Then the resin was washed with water three times and dried under reduced pressure. For ICP-AES analysis, the resin was treated with HNO_3 at 100 °C for 4 h.

Suzuki–Miyaura coupling reaction; general procedure

Into a 10 mL glass vial were placed aryl halide (0.5 mmol), phenyl- or 4-methoxy phenylboronic acid (0.65 mmol), base (1 mmol), Pd-loaded catalyst **4** (0.2 mol% Pd) and EtOH/water (1 : 1) (2 mL), and a magnetic stirrer was added. The vial was sealed, placed into the microwave cavity and the reaction mixture subjected to microwave irradiation of 60 W at 120 °C for 5 min. After completion, the reaction vessel was cooled to room temperature, opened and the contents were extracted with dichloromethane three times. The organic fraction was dried with magnesium sulfate, filtered and the solvent was removed under reduced pressure. The crude products were purified by chromatography on a silica gel column. All products were identified by ^{1}H NMR.

Biphenyl (entries 1, 10 and 11):[33] ^{1}H NMR: δ 7.60 (dt, J = 8.1, 1.5 Hz, 4H), 7.48–7.40 (m, 4H), 7.35 (ddd, J = 7.0, 2.2, 1.1 Hz, 2H).

4-Acetylbiphenyl (entry 2):[33] ^{1}H NMR: δ 8.05–8.03 (m, 2H), 7.7–7.69 (m, 2H), 7.64–7.62 (m, 2H), 7.49–7.46 (m, 2H), 7.42–7.39 (m, 1H), 2.64 (s, 3H).

4-Biphenylcarbaldehyde (entry 3):[34] ^{1}H NMR: δ 10.06 (s, 1H), 7.97–7.5 (m, 2H), 7.77–7.75 (m, 2H), 7.64 (ddd, J = 4.1, 3.2, 1.8 Hz, 2H), 7.50–7.47 (m, 2H), 7.43–7.41 (m, 1H).

4-Methoxybiphenyl (entries 13, 22 and 23):[33] ^{1}H NMR: δ 7.54 (dd, J = 12.6, 8.3 Hz, 4H), 7.42 (t, J = 7.6 Hz, 2H), 7.33–7.24 (m, 1H), 6.99 (t, J = 5.8 Hz, 2H), 3.85 (s, 3H).

The ^{1}H NMR data of the other products were consistent with published data: (entry 4,[35] entries 5 and 17,[36] entries 6, 7, 8, 12, 20, 21 and 24,[33] entries 9, 18 and 19,[34] entries 14,[37] 15[38] and 16[39]).

Catalyst stability and recycling

Iodobenzene (0.5 mmol), 4-methoxy phenylboronic acid (0.65 mmol), Na_2CO_3 (1 mmol), EtOH/H_2O (1 : 1) (2 mL), Pd-loaded catalyst **4** (0.2 mol% Pd) and a magnetic stirrer was added into a 10 mL glass vial. The vial was sealed and placed into the microwave cavity. The reaction was carried out at 120 °C for 5 min under microwave irradiation. After cooling to room temperature, the reaction contents were extracted with dichloromethane (5 mL × 2), and the solid polymer catalyst was filtered off and placed into another glass vial for the next reaction cycle. The filtered resins were reused five times for the same reaction. The yields were also determined by HPLC.

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