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Synthesis of ferrocenoyl amino acid derivatives via homogeneous catalytic aminocarbonylation

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Abstract

Palladium-catalysed aminocarbonylation of iodoferrocene with amino acid esters as nucleophiles results in the selective formation of *N*-ferrocenoyl amino acid esters in the presence of Et_3N as the base. At the same time, the use of DBU leads to the formation of new *N*-ferrocenylglyoxyl amino acid derivatives with reasonable selectivity. In the latter reactions two new side products, formed via acylation of DBU, were also isolated and characterised.

Keywords: Ferrocene; Amino acids; α-Ketoamides; Carbonylation; Pd-catalysts

1. Introduction

In the past few years, numerous electrochemical biosensors have been developed for the analytical determination of biological analytes [1–4]. Good stability and favourable electrochemical properties makes ferrocene derivatives good candidates for such purposes. Ferrocene-derived devices are used as anion and cation sensors [5,6], DNA biosensors [7–9] and as mediators of the electron transfer between active sites of oxidoreductases (e.g., glucose oxidase [10]) and electrodes. As a result, several methods have been reported for the synthesis of ferrocene-linked biomolecules, especially amino acids and peptides [11–18].

Although iodoferrocene has been used as substrate for the synthesis of protected ferrocenyl-alanine ((2-amino-2-carboxyethyl)ferrocene) derivatives via homogeneous catalytic coupling reactions [19,20], to the best of our knowledge there is no precedence for the synthesis of *N*-ferrocenoyl amino acids with palladium-catalysed aminocarbonylation.

Carbonylation of aryl halides in the presence of primary and secondary amines is a well-explored method for the selective synthesis of aryl amides and aryl ketoamides [21–23]. According to our recent results, amino acid esters can be successfully used as nucleophiles in the aminocarbonylation of iodobenzene and alkenyl iodides [24]. Therefore, as the next step in our series of investigations concerning the carbonylation reactions of iodoferrocene derivatives [25–28], the aminocarbonylation of iodoferrocene in the presence of amino acid esters has been explored.

In this reaction, *N*-ferrocenoyl amino acids can be synthesised via acylation of amino acid esters with the acyl-palladium intermediate formed in situ from the palladium precursor, carbon monoxide and iodoferrocene. As iodoferrocene can be readily produced from ferrocene via lithiation [29], this method is a good alternative of the generally used DCC/HOBt protocol starting from ferrocenecarboxylic acid [11–15].

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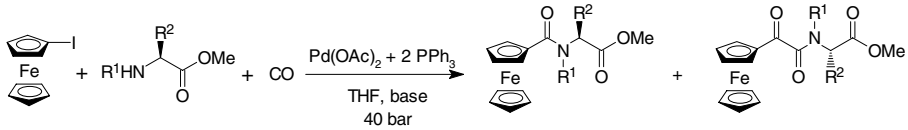
In the present work, it is also shown that in the aminocarbonylation of iodoferrocene, by the proper choice of the reaction conditions, new *N*-ferrocenylglyoxyl amino acid derivatives can also be obtained.

2. Results and discussion

2.1. Carbonylation of iodoferrocene in the presence of methyl glycinate

Our previous results concerning the aminocarbonylation of iodoferrocene with secondary amines [25,26] and that of iodobenzene with amino acid esters [24] demonstrated that both amide- and α-ketoamide-type products could be obtained in these reactions depending on the reaction conditions. Optimal conversion was achieved at 40–50 bar CO pressure. The use of relatively low temperatures (40–60 °C) favoured the formation of α-ketoamides, while amides were formed with high selectivity at 100 °C in most cases.



		R^1	R^2			
1	**2a**	H	H	(Gly-OMe)	**3a-e**	**4a-e**
	2b	H	CH_3	(L-Ala-OMe)		
	2c	H	CH_2Ph	(L-Phe-OMe)		
	2d	H	$(CH_2)_2SCH_3$	(L-Met-OMe)		
	2e	$-CH_2CH_2CH_2-$		(L-Pro-OMe)		

(1)

Accordingly, iodoferrocene (**1**) was reacted with a fivefold excess of methyl glycinate in the presence of an in situ Pd-catalyst (5 mol%) in an autoclave under CO pressure (Eq. (1)). At 100 °C and 40 bar CO pressure, complex **1** could be converted selectively into **3a** (Table 1).

However, contrary to the reactions of iodobenzene [24], the decrease in the temperature led to just a small increase in the ratio of **4a** to **3a** (but **3a** still remained the prevailing product). As a favourable effect of DBU on α-ketoamide formation has been reported by Inoue and co-workers [30], the carbonylation reaction has been carried out in the presence of various amounts of DBU (Runs 3–6, Table 1). As a result, the α-ketoamide/amide ratio has been increased, but the total yield observed by GC decreased simultaneously in spite of the total conversion of iodoferrocene in each case. It should be noted that the low values of GC yields are due to three facts according to the further investigations described in Section 2.2: (i) the formation of side products **8a**,**b**; (ii) decomposition of amide and ketoamide derivatives in the presence of DBU under non-inert conditions; and (iii) partial decomposition of the products in the presence of DBU in the inlet of the gas chromatograph.

2.2. Synthesis of N-ferrocenoyl- and N-ferrocenylglyoxyl amino acid esters

Based on the results described above, reaction conditions corresponding to Runs 1 and 4 in Table 1 were chosen for the synthesis of amides **3a**–**e** and ketoamides **4a**–**e**, respectively (Eq. 1, Table 2). The products were isolated and purified by column chromatography.

N-Ferrocenoyl amino acid esters **3a**–**e** were produced in excellent yields using Et_3N as base (Table 2, Runs 1, 4, 7, 10, and 13). However, in the presence of DBU only moderate amounts of the *N*-ferrocenylglyoxyl derivatives **4a**–**e** could be isolated (Runs 2, 5, 8, 11, and 14). According to the analysis of the reaction mixtures by

Table 1
Carbonylation of iodoferrocene in the presence of methyl glycinate (**2a**)[a]

Run	Base	Base/Fc–I (mol/mol)	*T* (°C)	Run time (h)	Conversion[b] (%)	Yield[b] (%)	
						3a	**4a**
1	Et_3N	6	100	8	100	100	0
2	Et_3N	6	60	8	82	73	9
3	DBU	6	100	8	100	58	4
4	DBU	8	100	8	100	9	32
5	DBU	8	60	12	99	8	13
6	DBU	10	100	2	100	4	12

[a] 40 bar CO.
[b] Determined by GC with eicosane as internal standard.

Table 2
Carbonylation of iodoferrocene in the presence of amino acid esters (**2a**–**2e**)[a]

Run	Nucleophile	Base	Conversion[b] (%)	Isolated yield (%)		
				3	**4**	**8a** + **8b**
1	**2a**	Et_3N[c]	100	94	–	–
2	**2a**	DBU[d]	99	1	25	2
3	**2a**	DBU[e]	99	1	89	5
4	**2b**	Et_3N[c]	92	87	–	–
5	**2b**	DBU[d]	99	17	20	14
6	**2b**	DBU[e]	99	23	28	21
7	**2c**	Et_3N[c]	85	81	–	–
8	**2c**	DBU[d]	97	2	10	29
9	**2c**	DBU[e]	96	15	26	42
10	**2d**	Et_3N[c]	81	75	–	–
11	**2d**	DBU[d]	98	3	17	19
12	**2d**	DBU[e]	98	4	37	32
13	**2e**	Et_3N[c]	90	84	–	–
14	**2e**	DBU[d]	100	32	29	3
15	**2e**	DBU[e]	100	32	30	19

[a] Reaction conditions: 40 bar CO, 100 °C, amino acid ester/**1** = 5, 8 h.
[b] Determined by GC with eicosane as internal standard.
[c] Et_3N/**1** = 6.
[d] DBU/**1** = 8.
[e] DBU/**1** = 8, during purification removal of DBU was carried out under inert conditions.

TLC, in addition to the expected products **3a**–**e** and **4a**–**e**, considerable amounts of a pair of ferrocene derivatives were also formed. The same novel compounds were obtained in all cases when DBU was used, irrespective of the amino acid ester nucleophile.

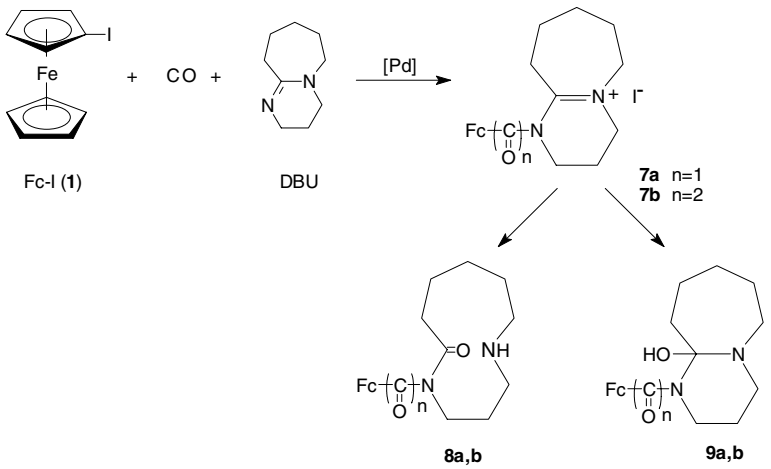
These compounds were also isolated and their structure were determined using various spectroscopic methods (^1H, ^{13}C NMR, correlation spectra (^1H–^1H and ^1H–^{13}C), MS and IR). The NMR spectra of these derivatives have shown the formation of two very similar ferrocene derivatives. In the ^{13}C NMR spectra of these compounds eight singlets appeared in the 20–50 ppm region corresponding to methylene groups. Two (at 177.2 and 170.2 ppm) and three further signals (at 190.9, 176.5 and 162.1 ppm) in the 160–190 ppm region were assigned to two DBU-derived amide- and ketoamide-type products, respectively.

According to the literature, DBU can be acylated with dimethyl- or dibenzyl carbonate [31,32] or can be alkylated with alkyl halides [33] leading to the corresponding salts. However, in the alkylation reaction a side product, formed by the opening of the bridge of the bicyclic compound, was also obtained. Another possibility is the hydrolysis of the salt leading to a hydroxy derivative, as it was observed for a reaction of DBU that took place in the coordination sphere of ruthenium [34]. Considering these observations, the carbonylation reaction of iodoferrocene with DBU as nucleophile may lead to products **8a**,**b** or **9a**,**b** (Scheme 1).

The appearance of the two and three singlets in the 160–190 ppm region of the respective ^{13}C NMR spectra unambiguously show the formation of **8a** and **8b**. The high downfield shift of the NH protons (7.38 ppm for **8a** and 8.00 ppm for **8b**) is probably due to the formation of a hydrogen bond between the NH and the 2-CO group.

It should be noted that when carbonylation of iodoferrocene is carried out in the presence of DBU alone (i.e., DBU itself serves as a nucleophile), without the addition of any amino acid esters, **8a** is the main product and **8b** is formed in traces only. On the contrary, in the reactions with amino acid esters, **8b** can be produced with good selectivity.

A careful examination of the reaction mixtures containing DBU showed the decomposition of amide and ketoamide type products (except **3e** and **4e**) in air. As no decomposition was observed in the THF solutions of the isolated compounds, in the further experiments the first step of the purification procedure, the removal of DBU was carried out under inert conditions. As a result, isolated yields were improved considerably in most cases (Table 2, Runs 3, 6, 9, 12, and 15).



Scheme 1. Possible products of carbonylation of iodoferrocene in the presence of DBU.

It should be noted that decomposition of amides and ketoamides in the presence of DBU was also observed under the conditions of gas chromatography. The decrease of the GC-yields with increasing amount of DBU as shown in Table 1. Runs 3–6 is partly due to this breakdown. At the same time, no decomposition of **3a**–**3d** and **4a**–**4d** took place in the absence of DBU. The starting material, iodoferrocene was inert both in the presence and in the absence of DBU.

The *N*-ferrocenylglyoxyl derivative **4a** can be produced with high selectivity using methyl glycinate as the nucleophile (Table 2.). DBU successfully competes with the other amino acid esters in the reaction with the acylpalladium intermediate, which results in the formation of considerable amounts of **8a**,**b**.

The α-ketoamide/amide ratio (**4/3**) is the lowest in the reaction of methyl L-prolinate. This is in accordance with our previous results that have shown that ketoamide formation is not favoured in the reactions of the sterically hindered nucleophiles [26].

3. Conclusion

N-Ferrocenoyl amino acid esters can be produced in high yields via palladium-catalysed aminocarbonylation starting from iodoferrocene and amino acid methyl esters. The use of an excess of DBU favours the formation of the α-ketoamides, and the corresponding *N*-ferrocenylglyoxyl amino acid esters can be obtained with moderate to good selectivity. In the latter reactions, the formation of two new ferrocene derivatives, obtained by the acylation of DBU by the acyl-palladium intermediate, can also be observed.

4. Experimental

The ^{1}H and ^{13}C NMR spectra were recorded on a VARIAN INOVA 400 spectrometer at 400 and 100.58 MHz, respectively. GLC analyses were carried out with a HP-5890/II gas chromatograph using a 15 m HP-5 column. Infrared (IR) spectra were recorded in KBr pellets using a Specord-IR 75 instrument. GC-MS measurements were performed with a Hewlett–Packard 5971A GC-MSD using HP-1 column.

4.1. Carbonylation of iodoferrocene (*1*)

Iodoferrocene (0.5 mmol), $Pd(OAc)_2$ (0.025 mmol), PPh_3 (0.05 mmol), the hydrochloride of the amino acid ester (2.5 mmol), Et_3N or DBU (as indicated in Tables 1 and 2) and THF (12.5 mL) were transferred under an inert atmosphere into a stainless steel autoclave. It was charged with carbon monoxide and heated with stirring in an oil bath.

During the work-up of the reaction mixture the volatile components were removed in vacuo. The residue was dissolved in toluene (20 mL), washed with 5% H_3PO_4 (20 mL), saturated aqueous $NaHCO_3$ (20 mL) and brine (20 mL) and dried over Na_2SO_4.

Alternatively, toluene (20 mL) was added to the reaction mixture after the completion of the reaction and the first washing with 5% H_3PO_4 (20 mL) was carried out under an inert atmosphere. After that, the procedure was the same as above.

The products were separated after removal of the solvent by column chromatography (aluminium oxide, toluene/EtOAc = 3/1 for **3a**–**e** and **4a**–**e**, EtOAc/MeOH = 10/1 for **8a**,**b**). The structures of the isolated compounds were determined by ^{1}H, ^{13}C NMR and IR spectroscopy, elemental analysis, and MS.

Physical properties and spectra of **3a**–**c** corresponded well to the literature data [35].

4.1.1. *N*-Ferrocenoyl-L-methionine methyl ester (*3d*)

^{1}H NMR($CDCl_3$) δ: 6.45 (brs, 1H, NH); 4.85 (m, 1H, CH); 4.71 (brs, 1H, Cp-2); 4.65 (brs, 1H, Cp-5); 4.34 (brs, 2H, Cp-3,4); 4.21 (s, 5H, Cp); 3.77 (s, 3H, OCH_3); 2.55 (t, 7 Hz, S–CH_2); 2.3-1.9 (m, 2H, CH_2); 2.12 (s, 3H, S–CH_3). IR (KBr (cm^{-1})): 3268; 1744; 1625; 1533. MS (*m*/*z*/rel. int.): 375 (M^+)/69; 213/100; 185/62; 129/37; 121/20; 56/25. Anal. Calc. for $C_{17}H_{21}FeNO_3S$ (375.27): C, 54.41; H, 5.64; N, 3.73. Found: C, 54.22; H, 5.82; N, 3.61%. Yield: 75%.

4.1.2. *N*-Ferrocenoyl-L-proline methyl ester (*3e*)

^{1}H NMR($CDCl_3$) δ: 4.85 (brs, 1H, Cp-2); 4.69 (brs, 1H, Cp-5); 4.61 (brs, 1H, CHCOO); 4.34 (brs, 2H, Cp-3,4); 4.25 (s, 5H, Cp); 3.94 (m, 1H, NCH_a); 3.81 (m, 1H, NCH_b); 3.76 (s, 3H, OCH_3); 2.2 (m, 2H, CH_2); 1.98 (m, 2H, CH_2).^{13}C NMR($CDCl_3$) δ:173.0; 169.6; 71.3; 70.5; 70.2; 69.7; 60.1; 52.1; 48.3; 28.7; 25.6. IR (KBr (cm^{-1})): 1740; 1590. MS (*m*/*z*/rel. int.): 341 (M^+)/79; 213/100; 185/45; 129/45; 121/47; 56/25. Anal. Calc. for $C_{17}H_{19}FeNO_3$ (341.19): C, 59.85; H, 5.61; N, 4.11. Found: C, 60.02; H, 5.50.; N, 4.23%. Yield: 84%.

4.1.3. *N*-Ferrocenylglyoxyl-glycine methyl ester (*4a*)

^{1}H NMR($CDCl_3$) δ: 7.64 (brs, 1H, NH); 5.31 (s, 2H, Cp-2,5); 4.71 (s, 2H, Cp-3,4); 4.24 (s, 5H, Cp); 4.14 (d, 5.6 Hz, 2H, CH_2); 3.79 (s, 3H, OCH_3). ^{13}C NMR($CDCl_3$) δ: 189.7; 169.5; 161.8; 74.5; 74.4; 74.3; 72.2; 70.6; 69.9; 52.5; 40.9 IR (KBr (cm^{-1})): 3430; 1740; 1680; 1650 MS (*m*/*z*/rel. int.): 329 (M^+)/79; 213/100; 185/82; 129/74; 121/47; 56/53. Anal. Calc. for $C_{15}H_{15}FeNO_4$ (329.14): C, 54.74; H, 4.59; N, 4.26. Found: C, 54.92; H, 4.71; N, 4.17%. Yield: 89%.

4.1.4. *N*-Ferrocenylglyoxyl-L-alanine methyl ester (*4b*)

^{1}H NMR($CDCl_3$) δ: 7.61 (d, 8 Hz, 1H, NH); 5.35 (brs; 1H, Cp-2); 5.25 (brs, 1H, Cp-5); 4.70 (brs, 2H,

Cp-3,4); 4.63 (quint., 8 Hz, 1H, CH); 4.22 (s, 5H, Cp); 3.78 (s, 3H, OCH_3); 1.51 (d, 8 Hz, 3H, –CH–CH_3). ^{13}C NMR($CDCl_3$) δ: 189.9; 172.5; 161.2; 74.5; 74.4; 74.3; 72.5; 71.8; 70.6; 52.6; 48.0; 18.0. IR (KBr (cm^{-1})): 3320, 1748; 1683; 1650. MS (*m*/*z*/rel. int.): 343 (M^+)/66; 213/100; 185/49; 129/41; 121/22; 56/13. Anal. Calc. for $C_{16}H_{17}FeNO_4$ (343.16): C, 56.00; H, 4.99; N,4.08. Found: C, 56.17; H, 5.12; N, 4.17%. Yield: 28%.

4.1.5. *N*-Ferrocenylglyoxyl-L-phenylalanine methyl ester (**4c**)

^{1}H NMR($CDCl_3$) δ: 7.24 (m, 5H, Ph); 6.50 (brs, 1H, NH); 5.25 (brs, 2H, Cp-2,5); 4.90 (m, 1H, CH); 4.68 (brs, 2H, Cp-3,4); 4.14 (s, 5H, Cp); 3.74 (s, 3H, OCH_3); 3.18 (m, 2H, CH_aH_b). ^{13}C NMR($CDCl_3$) δ: 189.7; 171.2; 161.1; 135.7; 129.2; 128.8; 127.3; 74.4; 74.3; 72.2; 72.1; 70.5; 53.1; 52.5; 38.0. IR (KBr (cm^{-1})): 3360, 1736; 1683; 1642. MS (*m*/*z*/rel. int.): 419 (M^+)/35; 213/71; 185/62; 129/70; 121/45; 91/100; 56/26. Anal. Calc. for $C_{22}H_{21}FeNO_4$ (419.26): C, 63.03; H, 5.05; N, 3.34. Found: C, 62.91; H, 5.14; N, 3.22%. Yield: 26%.

4.1.6. *N*-Ferrocenylglyoxyl-L-methionine methyl ester (**4d**)

^{1}H NMR($CDCl_3$) δ: 7.73 (d, 8 Hz, 1H, NH); 5.34 (s, 1H, Cp-2); 5.26 (s, 1H, Cp-5); 4.76 (m, 1H, CH); 4.71 (brs, 2H, Cp-3,4); 4.22 (s, 5H, Cp); 3.78 (s, 3H, OCH_3); 2.57 (t, 8 Hz, S–CH_2); 2.29–2.0 (m, 2H, CH_2); 2.11 (s, 3H, S–CH_3). ^{13}C NMR($CDCl_3$) δ: 189.7; 171.5; 161.4; 74.6; 74.5; 74.4; 72.5; 71.9; 70.6; 52.7; 51.4; 31.5; 30.1; 15.5. IR (KBr (cm^{-1})): 3360; 1740; 1683; 1642. MS (*m*/*z*/rel. int.): 403 (M^+)/85; 213/100; 185/53; 129/48; 121/24; 56/13. Anal. Calc. for $C_{17}H_{21}FeNO_3S$ (403.28): C, 53.61; H, 5.25; N, 3.47. Found: C, 53.84; H, 5.11; N, 3.59%. Yield: 37%.

4.1.7. *N*-Ferrocenylglyoxyl-L-proline methyl ester (**4e**)

^{1}H NMR($CDCl_3$) δ: 5.01 (brs, 2H, Cp-2,5); 4.73 (brs, 1H, CHCOO); 4.62 (brs, 2H, Cp-3,4); 4.3 (s, 5H, Cp); 3.8 (s, 3H, OCH_3); 3.65 (m, 2H, N–CH_2); 1.9-2.3 (m, 4H, CH_2-CH_2). ^{13}C NMR($CDCl_3$) δ: 189.2; 172.1; 164.5; 74.9; 73.8; 73.7; 70.6; 70.5; 70.4; 58.5; 52.4; 47.6; 29.0; 25.0. IR (KBr (cm^{-1})): 1744; 1665; 1634. MS (*m*/*z*/rel. int.): 369 (M^+)/43; 213/100; 185/49; 129/39; 121/26; 56/22. Anal. Calc. for $C_{18}H_{19}FeNO_4$ (369.20): C, 58.56; H, 5.19; N, 3.79. Found: C, 58.41; H, 5.23; N, 3.87%. Yield: 30%.

4.1.8. 1-Ferrocenoyl-1,8-diaza-cycloundecan-2-one (**8a**)

^{1}H NMR($CDCl_3$) δ: 7.38 (brs, 1H, NH); 4.80 (brs; 2H, Cp-2,5); 4.29 (brs, 2H, Cp-3,4); 4.19 (s, 5H, Cp); 3.54 (m, 2H, C(O)NCH_2); 3.34 (m, 4H, CH_2NCH_2); 2.59 (m, 2H, C(O)CH_2); 1.72 (m, 8H, 4CH_2). ^{13}C NMR($CDCl_3$) δ: 177.3; 170.2; 70.3; 70.2; 69.7; 68.3; 49.5; 44.7; 37.2; 34.7; 30.0; 28.4; 27.3; 23.5. IR (KBr (cm^{-1})): 1650; 1638. MS: 382 (M^+). Anal. Calc. for $C_{20}H_{26}FeN_2O_2$ (382.29): C, 62.84; H, 6.86; N, 7.33. Found: C, 62.97; H, 6.71; N, 7.41%.

4.1.9. 1-Ferrocenylglyoxyl-1,8-diaza-cycloundecan-2-one (**8b**)

^{1}H NMR ($CDCl_3$) δ: 8.00 (brs, 1H, NH); 5.29 (brs; 2H, Cp-2,5); 4.65 (brs, 2H, Cp-3,4); 4.20 (s, 5H, Cp); 3.47 (m, 2H, C(O)NCH_2); 3.34 (m, 4H, CH_2NCH_2); 2.55 (m, 2H, C(O)CH_2); 1.68 (m, 8H, 4CH_2). ^{13}C NMR($CDCl_3$) δ: 190.9; 176.5; 162.1; 74.8; 74.0; 72.1; 70.4; 49.7; 45.5; 37.2; 36.0; 30.0; 28.6; 27.8; 23.4. IR (KBr (cm^{-1})):. 1700; 1646; 1634. MS: 410 (M^+). Anal. Calc. for $C_{21}H_{26}FeN_2O_3$ (410.30): C, 61.48; H, 6.39; N, 6.83. Found: C, 61.32; H, 6.51; N, 6.75%.

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