olefin and carbon monoxide behind the center of the palladium focal point. The molar ratio of $AlCl_3$ in the catalytic system also strongly affects on the yield of the target products. An increase in the $PdCl_2(PPh_3)_2$–$PPh_3$–$AlCl_3$ ratio from 1:8 to 1:9 led to an increase in the yield of target products from 72.6 to 80.7 %; a further increase in the excess of $AlCl_3$ reduces the yield of the target product. No solvents were used in this reaction, and the ratio of the starting reagents affected the product yield. With the ratio $[C_8H_{10}]:[C_2H_5OH]$ = 661:435, the product yield is 72.3 %; further reduction to $[C_8H_{10}]:[C_2H_5OH]$ = 435:435 gives the highest product yield of 80.7 %, but further reduction to $[C_8H_{10}]:[C_2H_5OH]$ = 217.5:435 reduces the yield of the target product.

Table 1

**Hydroethoxycarbonylation of cyclohexene in the presence of the $PdCl_2(PPh_3)_2$–$PPh_3$–$AlCl_3$ system**

| Exp. no. | $[C_8H_{10}]:[C_2H_5OH]$ | $[PdCl_2(PPh_3)_2]:[PPh_3]:[AlCl_3]$ | $T$, °C | $P_{CO}$, MPa | τ, h | Product yield, % |
|---|---|---|---|---|---|---|
| 1 | 661:435 | 1:6:9 | 120 | 2.5 | 5 | 72.3 |
| 2 | 435:435 | 1:6:9 | 120 | 2.5 | 5 | 80.7 |
| 3 | 217.5:435 | 1:6:9 | 120 | 2.5 | 5 | 55.6 |
| 4 | 435:435 | 1:6:8 | 120 | 2.5 | 5 | 72.6 |
| 5 | 435:435 | 1:6:10 | 120 | 2.5 | 5 | 74.2 |
| 6 | 435:435 | 1:6:9 | 130 | 2.5 | 5 | 66.7 |
| 7 | 435:435 | 1:6:9 | 110 | 2.5 | 5 | 65.8 |
| 8 | 435:435 | 1:6:9 | 120 | 3.0 | 5 | 68.0 |
| 9 | 435:435 | 1:6:9 | 120 | 2.0 | 5 | 43.4 |
| 10 | 435:435 | 1:6:9 | 120 | 2.5 | 4 | 68.5 |
| 11 | 435:435 | 1:6:9 | 120 | 2.5 | 6 | 77.2 |

Thus, it was found that the three-component catalytic system $PdCl_2(PPh_3)_2$–$PPh_3$–$AlCl_3$, which contains $AlCl_3$ as a promoter in the carbonylation reaction of cyclohexene at a low carbon monoxide pressure (2.5 MPa), exhibits high catalytic activity. As a result, the following effective parameters were identified: $[C_6H_{10}]:[C_2H_5OH]:[Pd]:[PPh_3]:[AlCl_3]$ = 435:435:1:6:9, $P_{CO}$ = 2.5 MPa, T = 120 ºC, τ = 5 h. Under the developed optimal reaction conditions, the yield of ethyl ester of cyclohexanecarboxylic acid was 80.7%.

The study and identification of the fractionated product was carried out as mentioned above (experimental part), by gas chromatography method (shown in Figure 1). On the chromatogram we can observe a change in the value of the total ion current at the 19th minute of exposure, which, in turn, indicates the presence of the target product – ethyl ester of cyclohexanecarboxylic acid (at the 1st minute — unreacted ethanol; at the 3rd minute — unreacted cyclohexene).

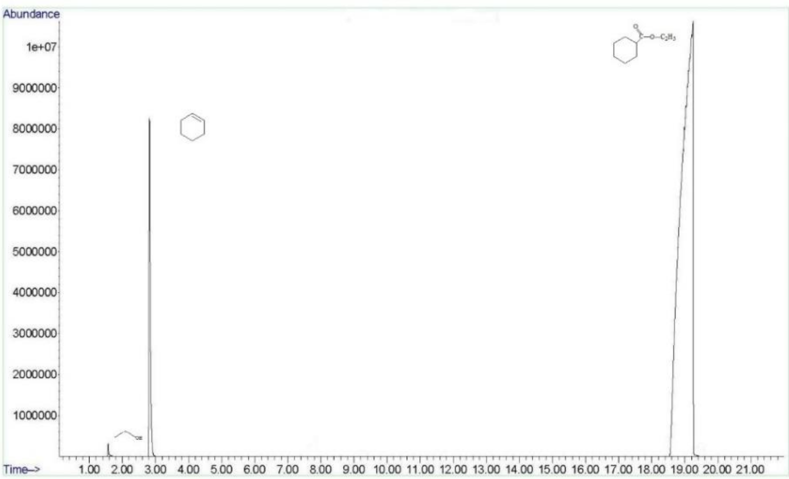


Figure 1. GC analysis of target product