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Study of the Process of Obtaining a Catalyst for the Synthesis of Benzene Based on Ethylene for the Production of Cyclohexane

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Abstract. Products of petrochemical industries are necessary for every modern person, thousands of enterprises in various industries. A result of the distillation of crude oil, cyclohexane ended up in naphtha, it was sent to catalytic reformers, where it was processed into benzene. Based on the results obtained in the laboratory for the synthesis of cyclohexane using ethylene, tests were carried out in the central laboratories Shurtan gas chemical complex. When analyzing the resulting product using gas chromatography, it was found thatabout 17.5% cyclohexane was formed. The technical parameters of the products (butadiene-1,3 and cyclohexane), synthesized by the staff of the National University of Uzbekistan and the Central Laboratory of the Shurtan Gas Chemical Complex, give approximate properties to imported cyclohexane at the JSC of the Shurtan Gas Chemical Complex.

Key words: Benzene, ethylene, cyclohexane, oil, catalyst, reaction of Dils-Alder

Products of petrochemical industries are necessary for every modern person, thousands of enterprises in various industries. These are varnishes, paints, solvents, polymers, detergents and cosmetics, medicines and much more. Interest in cyclohexane arose in 1938 in connection with the development of nylon by DuPont, which proposed the use of cyclohexane as the preferred raw material. After World War II, nylon production increased by 100% per year for some time, so that the cyclohexane contained in crude oil soon became insufficient. The standard crude oil that was supplied to US refineries at the time contained 1% cyclohexane. Moreover, since as a result of the distillation of crude oil, cyclohexane ended up in naphtha, it was sent to catalytic reformers, where it was processed into benzene. And subsequently, while many other substances were also converted to benzene by catalytic reforming, benzene became a good source of cyclohexane.

Cyclohexane (C_6H_{12}) is a cycle of six carbon atoms, each has 2 hydrogen atoms. It is similar to benzene, but it does not have double bonds. It is a colorless, water-insoluble and non-corrosive liquid with a pungent odor. It is combustible, like any product derived from oil; it is transported in tanks, tank trucks, barges and metal drums, which must have a red mark adopted for flammable liquids. The industry produces technical grade cyclohexane (purity 95% or 99%) and solvent cyclohexane (purity not less than 85%).

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Cyclohexane is also used in the Shurtan gas chemical complex as a solvent for the production of various grades of polyethylene. The polymerization catalysts are dissolved in pure cyclohexane and the ethylene polymerization process is carried out; after the process is completed, the cyclohexane is recovered by distillation and returned to the process

Based on the results obtained in the laboratory for the synthesis of cyclohexane using ethylene, tests were carried out in the centrallaboratoriesShurtan gas chemical complex. Under these conditions, several methods have been tried for the synthesis of ethylene-based cyclohexane.

Ethylene dimerizes to form butene-1. Triethylaluminum is used as a catalyst. The product formed in the process is rectified, and unreacted ethylene is returned to the original synthesis process. The cis-, trans-butene-2 and butadiene-1,3 formed in this process are also separated by distillation. Purified butene-1, cis-, trans-butene-2 is subjected to dehydrogenation to obtain butadiene-1,3. The catalyst is chromium (III) oxide; activated and acid-modified Navbakhor bentonite is used as a carrier. The resulting product is purified from butadiene-1,3, but unreacted butenes are returned to the process. Cyclohexene is obtained by a 4+2 cyclocoupling reaction (Diels-Alder reaction) based on 1,3-butadiene and ethylene obtained by distillation. The catalyst is aluminum chloride and aluminum oxide is used as a carrier. The product is purified by distillation, the resulting mixture of cyclohexane, cyclohexadiene and benzene is hydrogenated on a nickel catalyst to obtain cyclohexane. Hydrogen is used for hydrogenation formed during the synthesis of butadiene-1,3.

In the process of synthesis of butene-1 in the Shurtan gas-chemical complex, a mixture of various hydrocarbons is released as a secondary product. This mixture contains 38.0 - 42.0% butadiene-1,3. This by-product is currently sold as a low quality fuel. Butadiene-1,3 was isolated from this mixture by rectification. The resulting product is sent for the synthesis of cyclohexane, as in the first method.

Benzene is synthesized from ethylene. At the same time, activated and acid-modified Navbakhor bentonite was used as a carrier, and chromium oxide (III) was used as a catalyst. From the mixture, benzene is purified by distillation-extraction (the extractant is dimethyl sulfoxide) and rectification. Next, hydrogenation of benzene is carried out, as in the first method, and cyclohexane is obtained.

According to the first proposed method, gaseous products obtained during the synthesis of butene-1 based on ethylene on a granular catalyst impregnated with bentonite, chromium (III) oxide at atmospheric pressure at a temperature of 550–600°C, the catalyzate was reanalyzed by gas chromatography with a yield of 8.74 mol. % butadiene-1.3. The resulting product is separated by distillation with cooling, and the remaining gas mixture is returned to the synthesis.

Table 1

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Data File C:\HPCHEM\1\DATA\151221\SIG10004.D Sample Name: Exsprement-5

Exsprement 5 Time:11:30. Data:15.12.2021

```
Injection Date    : 15.12.21 16:03:03
Sample Name       : Exsprement-5                    Vial :   1
Acq. Operator     : Eshonqulov G                     Inj :   1
                                                Inj Volume : Manually
Acq. Method       : C:\HPCHEM\1\METHODS\DEF_UPC4.M
Last changed      : 15.12.21 16:35:46  by Eshonqulov G
                    (modified after loading)
Analysis Method   : C:\HPCHEM\1\METHODS\DEF_UPC4.M
Last changed      : 22.12.21 14:57:45  by Niyazov SH
                    (modified after loading)
```

Area Percent Report

```
Sorted By             :      Retention Time
Calib. Data Modified  :      22.12.21 14:53:39
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FID1 A,
```

Peak #	RetTime [min]	Sig	Type	Area [pA*s]	Area %	Name
1	3.090	1		0.00000	0.00000	propane
2	3.532	1		0.00000	0.00000	iso-butane
3	3.636	1		0.00000	0.00000	n-butane
4	4.400	1		0.00000	0.00000	neo-pentane
5	4.440	1		0.00000	0.00000	iso-pentane
6	4.720	1		0.00000	0.00000	Acetone
7	4.819	1		0.00000	0.00000	n-pentane
8	5.024	1	PB	12.67662	0.00138	?
9	5.523	1		0.00000	0.00000	2,2-dimethyl butane
10	6.479	1		0.00000	0.00000	2,3-dimethyl butane
11	6.749	1	PV	40.56224	0.00442	?
12	6.917	1	VB	8.63659	0.00094	2-methyl pentane
13	7.170	1		0.00000	0.00000	3-methyl pentane
14	7.199	1		0.00000	0.00000	MEK
15	8.043	1	PB	33.92939	0.00369	n-hexane
16	8.400	1		0.00000	0.00000	ic7
17	8.637	1		0.00000	0.00000	ic7
18	8.854	1		0.00000	0.00000	ic7
19	9.148	1	PB	120.69862	0.01314	?
20	10.812	1		0.00000	0.00000	ic7
21	10.920	1		0.00000	0.00000	ic7
22	11.012	1		0.00000	0.00000	ic7
23	11.064	1	BV	6.81054e5	74.13089	benzene
24	11.277	1		0.00000	0.00000	ic7
25	11.343	1	VV	2.36650e5	25.75864	cyclohexane
26	11.393	1	VV	8.41990	0.00092	ic7
27	11.537	1	VB	34.23016	0.00373	ic7
28	11.757	1	BV	13.83569	0.00151	?
29	11.854	1	VV	13.48413	0.00147	?
30	11.948	1	VB	11.55218	0.00126	n-heptane
31	12.387	1	BB	12.04707	0.00131	?
32	12.877	1		0.00000	0.00000	methyl cyclohexane
33	12.900	1		0.00000	0.00000	ic8
34	12.947	1		0.00000	0.00000	ic8
35	13.256	1	BB	218.41940	0.02377	ic8
36	13.727	1	BB	17.44350	0.00190	ic8

GC-3 22.12.21 14:57:46 Niyazov SH Page 1 of 2

Table 2

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Data File C:\HPCHEM\1\DATA\091221\SIG10004.D

Eksperiment Butadiyen-1,3 Tim:16:24. Data:09.12.20
21

```
================================================================================
Injection Date  : 09.12.21 17:29:09
Sample Name     :                                          Vial :   1
Acq. Operator   : Islomberdiev S                           Inj :    1
                                                  Inj Volume : Manually
Method          : C:\HPCHEM\1\METHODS\S1401MOL.M
Last changed    : 09.12.21 18:18:42  by Islomberdiev S
                  (modified after loading)
================================================================================
                        Normalized Percent Report
================================================================================

Sorted By             :      Retention Time
Calib. Data Modified  :      09.12.21 18:18:40
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FID1 A,

RetTime Sig Type      Area      Amt/Area      Norm     Grp   Name
 [min]              [pA*s]                    %
-------|--|------|----------|----------|----------|--|---------------
  3.296  1 VV      396.49097 4.10703e-3   1.834888     Methane
  3.376  1 VV      220.24570 2.16148e-3   0.536423     Ethylene
  3.425  1 VV      102.88946 2.13306e-3   0.247299     Ethane
  3.780  1 VV      849.08209 1.60514e-3   1.535715     Propylene
  3.825  1 VV       47.05563 1.47295e-3   0.078100     Propane
  4.074  1 VV        7.05199 1.38672e-3   0.011019     MethylAcetylene
  4.100  1 VV        3.14275 1.62506e-3   0.005755     Propadiene
  4.531  1 VV        2.89980 1.24835e-3   0.004079     iso Butane
  5.083  1 VV S   2.37978e4  1.29791e-3  34.804184     1 Butene
  5.168  1 VV S   4334.66260 1.78841e-3   8.735163     1,3-Butadiene
  5.301  1 BB X    190.83209 1.12591e-3   0.242105     N-Butane
  5.602  1 VV S   1.92096e4  1.22523e-3  26.520774     Tr-2-Butene
  6.050  1 VV S   1.70335e4  1.18163e-3  22.679507     Cis 2-Butene
  6.500  1            -          -          -         isobutylen
 22.123  1 MM     2666.46216 9.20260e-4   2.764991     C5+

Totals :                                 100.000000

 Results obtained with enhanced integrator!
2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)
Warning : Calibrated compound(s) not found

================================================================================
                          *** End of Report ***
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By passing a mixture of purified 1,3-butadiene and ethylene through a catalyst prepared by impregnating aluminum chloride into activated alumina at a temperature of 500-600°C. The product obtained by the Diels-Alder reaction at a temperature of 280-300°C, hydrogenation with hydrogen at a pressure of 2.0-3 MPa. When analyzing the resulting product using gas chromatography, it was found thatabout 17.5% cyclohexane was formed. Chromatographicanalysisthe product of hydrogenation of pure benzene on a nickel catalyst at a pressure of 2.0-3.0 MPa at 250-280°C gives 25.76 mol.% of the product. The results of the analysis are presented in the following tables (Tables 1 and 2).

The technical parameters of the products (butadiene-1,3 and cyclohexane), synthesized by the staff of the National University of Uzbekistan and the Central Laboratory of the Shurtan Gas Chemical Complex, give approximate properties to imported cyclohexane at the JSC of the Shurtan Gas Chemical Complex. Only for further optimization of this synthesis process, it is necessary to continue joint research to equate its technical parameters with imported cyclohexane.

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