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TECHNOLOGY FOR THE SYNTHESIS OF 4′, 4′′-DI-(1-METHYL-1-HYDROXYETHYNYL)-DIBENZO-18-CROWN-6

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Abstract

The synthesis and technology process of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6 are proposed, the characteristics of the initial reagents and reaction products are given, the chemistry of the process with the most probable by-products and parallel reactions is described. The material balance of production is described, the parameters of the synthesis technology are studied, possible operational malfunctions and methods for their elimination are described, and analytical control of production is proposed.

Keywords: *4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6, technology, material balance, control*

The best available technology is a product production technology determined on the basis of modern achievements of science and technology and the best combination of criteria for achieving environmental protection, provided that it is technically possible to use it.

One of the most large-scale and widely used industrial products is acetylene. Applications of acetylene include the production of polyvinyl chloride (Ma X., Wei H., Luo Z., 2024, 917–9490, acetylenides (explosives) (Trujillo-Lemon M., 2025, 1176–1187), acetic acid (Shuhrat o‘g‘li. O. B., 2025, 182–186), aromatic hydrocarbons (Bedenko S. P., Dement’ev K. I., Maximov A. L., 2022)., 989–1026), solvents (Fromme T., Reichenberger S., Tibbetts K. M., Barcikowski S., 2024, 638–663), rubbers (Agbaba O., Trotus I. T., Schmidt W., Schüth F., 2023, 1819–1825), acetaldehyde and many others (Zhang Z., Nabera A., Guillén-Gosálbez G., Pérez-Ramírez J., 2025, 1–11).

The production technology of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6 was developed jointly with employees of the Department of Oil and Gas Industry Technology, Faculty of Oil and Gas, Tashkent State Technical University (Kozinskaya L., Mirkhamitova D., 2021, 18–21). A pilot plant was installed to test the process.

The process is periodic. Power is set. The developed process consists of the interaction of acetylene with 4′,4″-diacetyldibenzo-18-crown-6 in the presence of a solvent (diethyl ether, tetrohydrofuran, benzene, toluene, etc.) and a catalyst – powdered potassium hydroxide.

No solid waste is generated during the production of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6. A spent aqueous solution of caustic potassium is formed as a liquid waste, which is sent for disposal, and the gaseous waste is unreacted acetylene, which is sent to a flare.

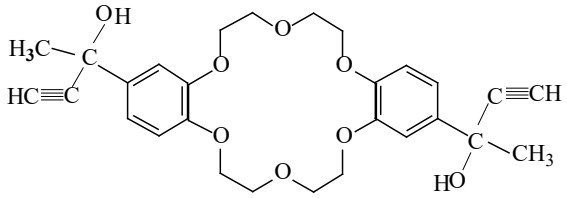
1. Characteristics of finished products

1.1. 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6

Amorphous white powder.

Empirical formula $C_{28}H_{32}O_8$

Structural formula



Molecular weight, c.u.	496
Melting point,°C	164–168
Solubility in: water	insoluble
organic solvents	dissolves well
chloroform	dissolves well
benzene	dissolves well
dimethyl sulfoxide	dissolves well

2. Characteristics of feedstock, materials and intermediate products

2.1. Acetylene, ethylene. C_2H_2; HC≡CH

Pure acetylene is a colorless gas with a faint ethereal odor.

Molecular weight, c.u.	26.04
Melting point,˚C	80.8
Boiling point, °C	83.8 (pressure 760 mm. Hg)
Sublimation temperature, °C	84.1

Table 1. *Characteristics of acetylene*

Temperature °C	0	10	20	30	40
pressure 0.5 atm	0.5833	0.5624	0.5430	0.5248	0.5879
pressure 1.0 atm	2.1716	0.1290	1.0805	1.0528	1.0285
pressure ë1.5 atm	1.7850	1.7000	1.6308	1.5898	1.5318

The auto-ignition temperature of acetylene at atmospheric pressure is 635 °C

Table 2. *Self-ignition temperature of acetylene-air mixtures*

Content in the mixture, % vol.	10	20	30	45–55
Temperature, °C	500	400	374	135

Critical temperature, ˚C	35.6
Critical pressure, atm.	61.6
Calorific value (00, 760 mm Hg)	12710–13377 kcal/m^3

Acetylene is highly soluble in many organic and inorganic solvents. The solubility of acetylene in water is measured by the number of its volumes soluble in 1 volume of water at 0 ˚Cand a partial pressure of acetylene of 760 mm Hg. Art., and is characterized by the following data.

Table 3. *The solubility of acetylene in water is measured by the number of its volumes soluble in 1 volume of water at 0 °C*

Temperature, °C	0	5	10	15	20	25	30	35	40	45
Solubility, m^3/m^3	1.75	1.52	1.32	1.16	1.03	0.93	0.85	0.77	0.71	0.65

Table 4. *Solubility of acetylene in organic solvents (pressure 760 mm Hg)*

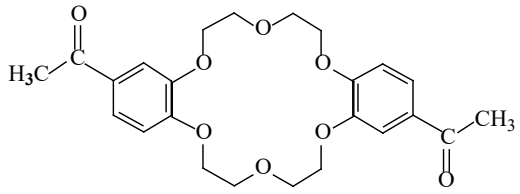
Solvent	Acetylene solubility, m^3/m^3					
	–20 °C	0 °C	20 °C	40 °C	60 °C	80 °C
Acetone	76	42	24	6.5	–	–
γ-Butyrolactone	–	19.5	11.5	6.1	4.4	–
Dimethylformamide	–	67.0	37.4	21.4	13.0	8.0
Methanol	40	20	11.2	6.0	–	–
N- methyl pyrrolidone	–	65	38.7	23	10	7

2.2. 4′,4″- diacetyldibenzo-18-crown-6

Amorphous white powder.

Empirical formula $C_{24}H_{28}O_8$

Structural formula

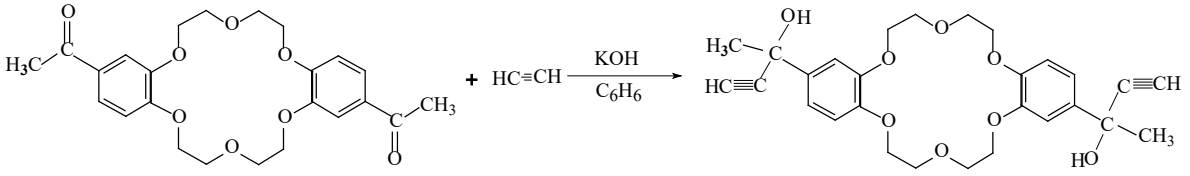


Molecular weight, c.u.	444
Melting point,0C	194–200
Solubility in: water	insoluble
organic solvents	dissolves well
chloroform	dissolves well
benzene	dissolves well
dimethyl sulfoxide	dissolves well

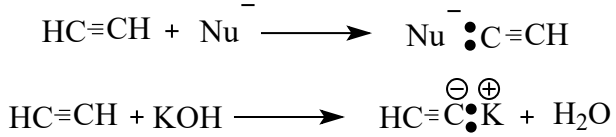
Chemistry of preparation of 4′,4″-di-(1-methyl-1-hydroxyethynyl) -dibenzo-18-crown-6

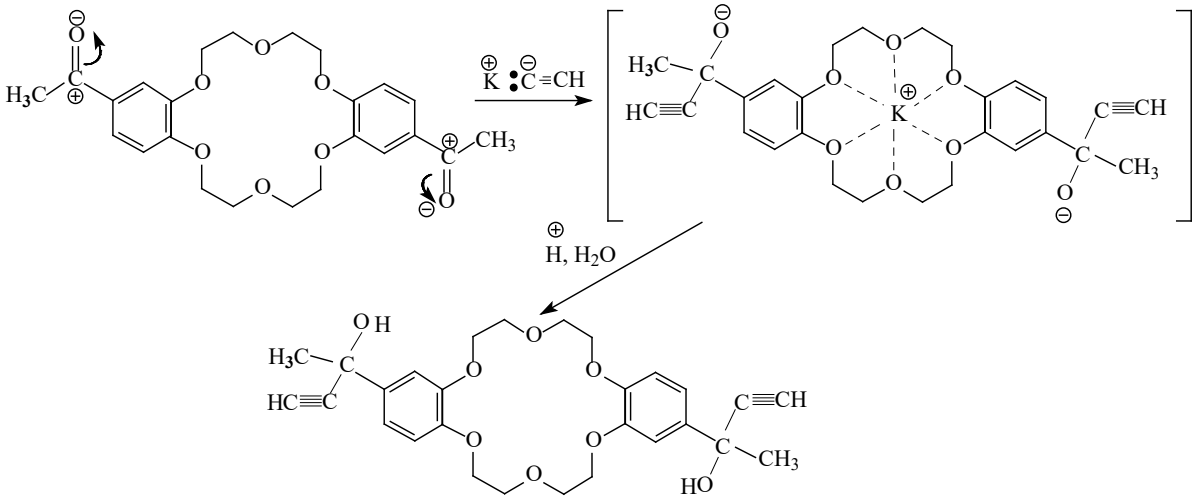
The reaction of the formation of acetylene alcohols in the presence of potassium hydroxide was discovered by A. Favorsky in 1905 (Favorsky A. E., 1905, 643–645).

The interaction of ketones with a suspension of powdered KOH in a solvent (ether, benzene, toluene, etc.) is carried out at a temperature of 0 °C+40 °C, a pressure of 0.4–0.9 MPa. The interaction of 4′,4″-diacetyldibenzo-18-crown-6 with acetylene was carried out according to the following scheme:

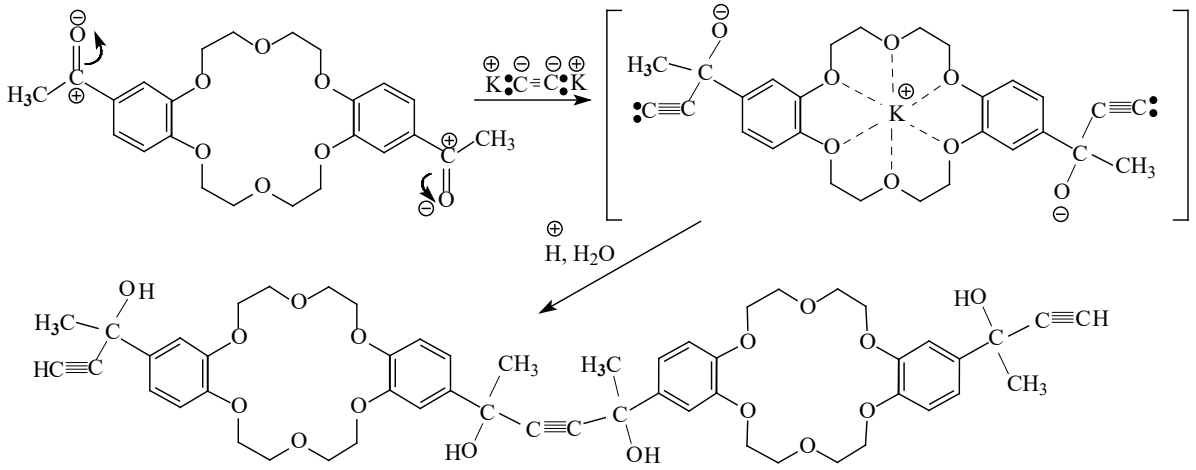


The mechanism of nucleophilic addition to the carbonyl group of the acetylenide ion formed upon deprotonation of the terminal alkyne has been proven (Reutov O. A., Kurts A. L., Butin K. P., 2021, 35–52). By analogy, the following mechanism for the formation of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown is presented –6:





It has been experimentally established that the amount of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6 formed per unit time is directly proportional to the concentration of potassium hydroxide. With an excess of acetylene, the reaction is pseudo-monomolecular with respect to potassium hydroxide. Changing conditions, using a slight excess of acetylene at a temperature of ~20 °C leads to the formation of acetylene 1,4-glycols:



The rate of formation of acetylene 1,4-glycols at a temperature of 20+22 °C significantly exceeds the rate of formation of tertiary acetylene alcohols. The yield reaches 80%. As the temperature increases, the process is completely directed towards the formation of 1,4-glycols.

Experimental part

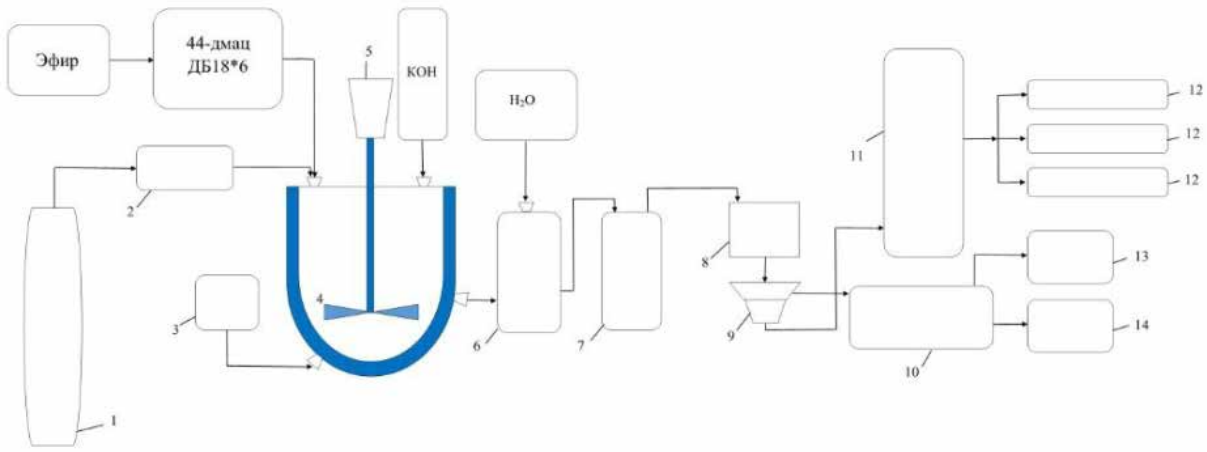
4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6. To 5.0 g (11.26 mmol) of 4′,4″-diacetyldibenzo-18-crown-6 in 200 ml of benzene, 63.06 g (112.6 mmol) of potassium hydroxide was added and heated for 20–30 min, then acetylene 630 ml (28.15 mmol), the mixture was boiled for 3 hours. The progress of the reaction was monitored by TLC on silufol in the system acetone: hexane, 2:1. Then hydrolysis was carried out in a weakly acidic medium, the precipitate was washed, purified by fractional crystallization from hexane, and dried in an oven at 40 °C. The compound was obtained in a yield of 2.35 g (42%), m.p. 164–168 °C.^{1}H-NMR ($CDCl_3$, 400 MHz) δ_H, mp: 1.82 (6H, s, α-C**H**$_3$), 2.65 (2H, s, -O**H**), 3.21 (2H, s, ≡C**H**), 3.87–4.26 (16H, m, α- and β-O-C**H**$_2$), 6.71–6.73 (2H, m, Ar-**H** 6′), 6.84 (2H, d, Ar-**H** 3′, *J*= 8,9 Hz), 6.87–6.89 (2H, d, Ar-**H** 5′, *J*= 6,3 Hz).^{13}C-NMR ($CDCl_3$, 100 MHz) δ, mp.: 33.10 (R-**C**H$_3$), 67.41 (–**C**-), 69.64–70.49–71.08 (β-α-O-**C**H$_2$), 73.10 (ArC≡**C**-), 87.20 (Ar**C**≡), 112.13 (Ar-**C**3′), 114.78–137.31 (Ar-**C**4′,5′,6′), 147.60–151.36 (Ar**C**-O-CH$_2$). Elemental analysis: found, %: C 67.93, H 6.44. Calculated, %: C 67.75, H 6.45 Gross formula: $C_{28}H_{32}O_8$.

Description of the technological process for the production of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6

Acetylene from the gas holder is supplied through a fire arrester to the lower part of the reactor. Acetylene consumption is measured by a gas meter. The reactor contains operating quantities of solvent, 4′,4″-diacetyldibenzo-18-crown-6 and potassium hydroxide.

4′,4″-diacetyldibenzo-18-crown-6 is saturated with acetylene at a temperature of 5–100C, which is created by cooled water supplied to the reactor jacket. Unreacted acetylene enters the reactor.

Picture 1. *Production flow diagram 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6*



1-cylinder with acetylene, 2-gas meter with pressure gauge, 3-thermocouple, 4-reactor, 5-mixer, 6-hydrolyzer, 7-separator, 8-stripping, 9-filtration, 10-crystallization, 11-distillation column, 12 – containers for fractions, 13 – drying, 14 – container for the finished product

During the interaction of 4′,4″-diacetyldibenzo-18-crown-6 acetylene into reactor 4 with constant mechanical mixing, diethyl ether is supplied from the container, and potassium hydroxide is supplied from the hopper. The contents are heated until the potassium hydroxide dissolves in the reaction mixture. After this, acetylene is continuously supplied from gas tank 1 into the reaction mixture through fire arrester 2 at a rate of 34.94 l/hour. 4 hours after cooling, the resulting catalyzate enters hydrolyzer 6 and passes through the stage of filter 9 and crystallization 10. The organic layer is collected in a rectification column 11 for further separation of the mixture in container 12. In this case, diethyl ether is sequentially determined. And the sediment of the target product is sent for drying 13 and collected in 14, 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6 at 155^{0}C, unreacted acetylene after purification was returned to the cycle.

Material balance of production of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6

60% DEE solution in terms of 1 ton of finished product

Table 5.

Consumption	Weight. kg	Coming	Weight. kg
1. Acetylene 100%	52.945	Solution of 4′,4′′-di-(1-methyl-1--hydroxyethynyl)-diben-zo-18-crown-6	1595.15

	Consumption	Weight. kg	Coming	Weight. kg
	Including C_2H_2 99%	52.416	Wt.h 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6 62.69%	1000
	N_2 0.3%	0.1588	DEE 40%	638,06
	O_2 0,1%	0.0529	N_2 0.0099%	0.1588
	CO_2 0,2%	0.1059	O_2 0.0033%	0.0529
2.	4′,4″-diacetyldibenzo-18-crown-6	895.104	CO_2 0.00664%	0.1059
	Incl. 4′,4″-diacetyldibenzo-18-crown-6 99.8%	904.145	H_2O 0.1133%	1.808
	H_2O 0.2%	1.808		
3.	DEE 100%	638.06		
	Total	**1595.15**	**Total**	**1595.15**

Calculation of acetylene consumption per 1 ton of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6

HC≡CH

$$\frac{1000}{496} = 2.016 \text{ k mole}$$

2.016 × 26 =52.416 kg 100% C_2H_2

Let's find 99% consumption C_2H_2

$$\frac{52.416}{0.99} = 52.945 \text{ kg } C_2H_2$$

of which 1% impurities by weight:

52.945 × 0.003 = 0.1588 kg N_2

52.945 × 0.001 = 0.0529 kg O_2

52.945 × 0.002 = 0.1059 kg CO_2

Acetylene volumetric flow:

$$\rho = 1.09 \quad V = \frac{52.945}{1.09} = 48.573\ m^3$$

Calculation of consumption of 4′,4″-diacetyldibenzo-18-crown-6 per 1 ton of finished product

2.016 kmol x 444 kg/kmol = 895.104 kg 100% 4′,4″-diacetyldibenzo-18-crown-6

consumption of 4′,4″-diacetyldibenzo-18-crown-6 99%:

$$\frac{895.104}{0.99} = 904.145 \text{ kg } 4',4''\text{- dia-}$$

cetyldibenzo-18-crown-6

0.2% impurity (moisture)

904.145 x 0.002 =1.808 kg moisture

DEE consumption 40% of the resulting solution:

52.945+904.145=957.09 kg of product

$$\frac{957.09}{0.6} = 1595.15 \text{ kg}$$

Consumption of 100% DEE:

19995.15–957.09=638.06 kg

Total: 52.945+904.145+638.06=1595.15 kg

Calculation of the concentration of the main and by-products:

$\frac{1000}{1595.15} \cdot 100 = 62.69\%$ 4′,4″- dia-cetyldibenzo-18-crown-6

$$\frac{638.06}{1595.15} \cdot 100 = 40\% \text{ DEE}$$

$$\frac{0.1588}{1595.15} \cdot 100 = 0.009955\% \ N_2$$

$$\frac{0.0529}{1595.15} \cdot 100 = 0.003316\% \ O_2$$

$$\frac{0.1059}{1595.15} \cdot 100 = 0.006639\% \ CO_2$$

$$\frac{1.808}{1595.15} \cdot 100 = 0.1133\% \ H_2O$$

Study of the technology parameters for the synthesis of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6:

— the technological process is carried out under atmospheric lighting;

— the total duration of the process is 10 hours, of which 2 hours are preparation and suspension; interaction of reagents with each other 4 hours, hydrolysis of intermediate products – 1 hour, separation 1 hour and fractional separation of the resulting products – 2 hours;

— for the initial three stages the temperature of the process is 0 °C, the fourth stage is 100 °C and for the rectification of the process 180 °C;

— the process is carried out periodically and continuously, and the supply of acetylene is 0.365 l/hour, 4′,4″-diacetyldibenzo-18-crown-6 0.294 l/hour, DEE 0.264 l/hour and KOH 0.112 kg/hour;

— the yield of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6 is 3.15 mol/l hour and the reaction rate is 0.28 mol/l. hour. In this case, the activation energy of the reaction is 8.05 kcal/mol;

— during the process, safety and environmental protection requirements are observed;

— it was established that the melting point of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6 is 164–168 °C, the structure was established according to IR,^{1}H- and^{13}C-NMR data – spectroscopy, the composition was determined by elemental analysis, and the purity was determined by GLC.

Industrial waste, wastewater and air emissions

The production of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6 produces liquid and solid waste. After rectification, liquid organic waste is returned to the cycle; the potassium hydroxide solution, after neutralization, is sent for disposal.

Table 6. *Technological standards*

No.	Name of stage and reagent flows	Operation time, hour	Temperature, °C	Pressure, MPa	Component ratio, mol	Conversion, %
	Formation of acetylene-KOH complex	1	0	atm	1:1	~100
	Synthesis 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6	4	34	atm	1:2.5	64–70

Table 7. *Production control and process control. Technological production control*

No.	Position number on the diagram	Parameter name and sampling location	Frequency and method of control	Standard and technical parameters	Installation location	Name and characteristics	Model type	Who controls
Formation of acetylene-KOH complex								
	R4	R4 reactor temperature control	Constantly	0 °C	On the R4 reactor on the CPU	Thermocouple GRHA, automatic potentiometer. Recorder showing GRHA measurement limits 0–600 °C intrinsically safe design	THA 0515 Ts2.821.71 102, KSP 44. Modification 41.540 50.540 50.008/41	Operator

No.	Position number on the diagram	Parameter name and sampling location	Frequency and method of control	Standard and technical parameters	Installation location	Name and characteristics	Model type	Who controls
Synthesis of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6								
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Table 8. *Analytical production control*

No	Name of process stages, sampling location or parameter changes	Controlled parameter	Frequency and method of control	Standards and technical indicators	Test method and controls	Who controls
1	Mixing acetylene with KOH	Acetylene content	1 time per shift	Not less than %	According to GOST 236–54	Laboratory assistant
2	Mixing ingredients in R4	Content of 4′,4″-diacetyldibenzo-18-crown-6	1 time per shift	Not less than %	Chromatographically	Laboratory assistant
3	Purity of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6 after recrystallization	Purity 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6	1 time per shift	Not less than %	By melting point	Laboratory assistant

Table 9. *Possible malfunctions and ways to eliminate them*

Problems	Possible causes of problems	Personnel actions and troubleshooting method
Formation of acetylene-KOH complex		
Reactor temperature rises	Increased coolant supply to R4	The supply of coolant to the R4 reactor has decreased
Synthesis of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6		
The content of the main substance in the R4 reactor has increased	The residence time of substances in the reactor has increased over 4 hours	Carry out hydrolysis and recrystallization of the main product

Environmental protection and basic rules for safe process management

The technological process for producing 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6 occurs using explosive and toxic substances – diethyl ether, acetylene, etc.

In accordance with the established fire safety category (A), classes of premises PUE (B-1g), as well as the category and condi-

tions of explosion hazard of operating mixtures. The installations must be operated in full compliance with the safety and industrial sanitation requirements set out in the "Rules and Standards for Safety and Industrial Sanitation for the Design and Operation of Fire and Explosion Hazardous Plants in the Chemical and Petrochemical Industry", "Sanitary Standards for the Design of Industrial Enterprises", "Rules for Construction" electrical installations."

The main condition for the safe conduct of the production process of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6 is strict adherence to workplace instructions, technological standards, safety regulations and fire safety regulations.

In order to ensure safe working conditions for maintenance personnel and protect equipment from destruction and fire, as well as reduce the consequences of accidents in production, the following measures are provided:

1. Installation for the synthesis of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6 located outside the building.
2. Automatic stroke control from the CPU.
3. Electric motors, electrical equipment, as well as automation and remote control devices installed in an explosive state.
4. To prevent the spread of diethyl ether combustion, fire arresters are installed on the lines.
5. Safety valves are installed on devices and pipelines where overpressure is possible.
6. Pipelines and devices with temperatures above 60^0C are insulated.

In addition to the above, it is necessary:

1. Have emergency supplies of serviceable filter and hose gas masks in the production area.
2. Before putting the equipment into operation, ensure the availability of raw materials, energy, water and materials. Check the installation locations of the plugs on the fittings of the devices and on the pipelines, the positions of the shut-off devices, the presence and serviceability of all necessary devices.
3. Load KOH wearing respirators and gloves.
4. Smoking and the use of open fire in the premises and on the territory of the workshop, except in specially designated areas, is prohibited.
5. Do not allow work in the presence of toxic and explosive products in the atmosphere of the premises in concentrations exceeding the permissible value.
6. Have first aid kits at work places with the necessary first aid supplies.

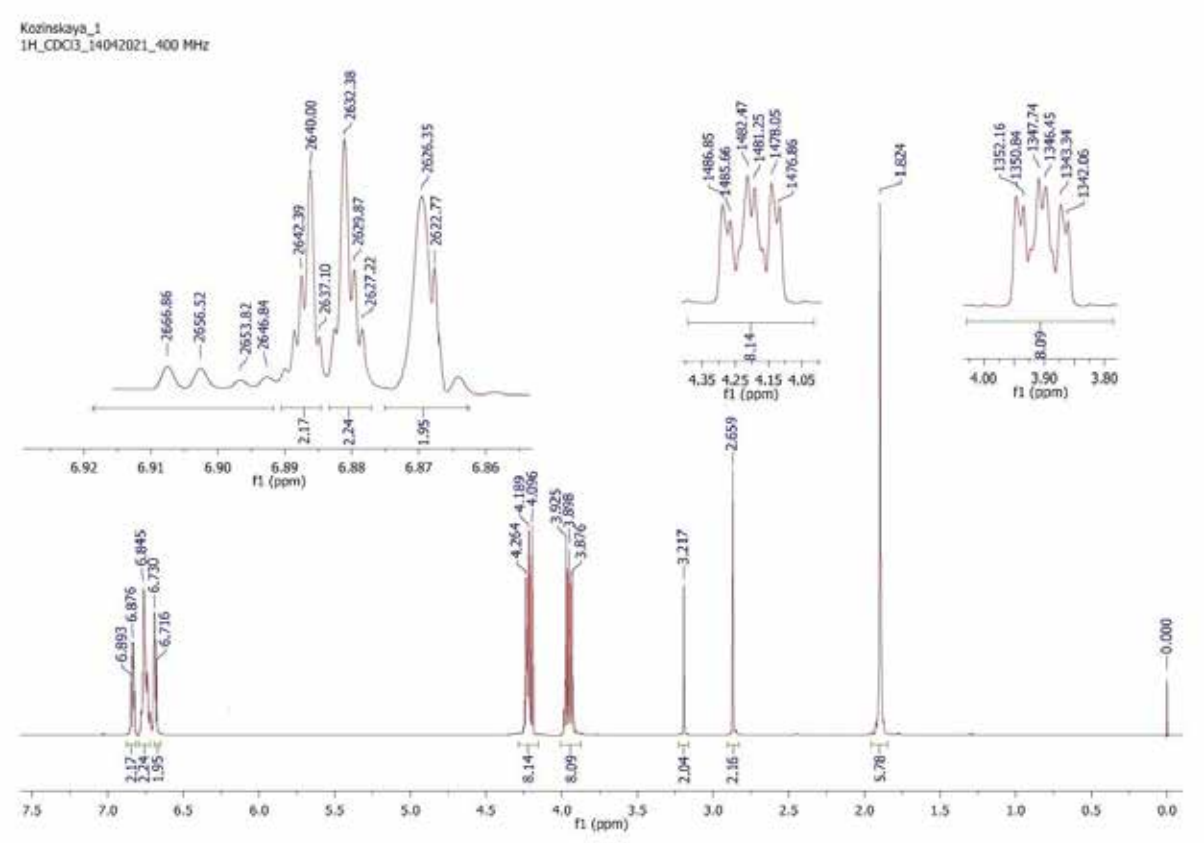
Table 10. *Specification of main process equipment*

Position number on the diagram	Equipment name	Quantity	Material, methods of protection	Specifications
1,2 R4	Solvent containers Reactor	2 1	Art. carbon. 12 × 18 H10 T	V=1m^3 V=2,5 m^{3,} P=0.-006, MPa=Pj=0.4 MPa, D $_{app}$=1400, D$_{rub}$=1500, H$_{sum}$=2406, N=5,5 kW, n=970 min^{-1}, U$_{сп}$=VZTU
2	Pump	1	12 × 18 H10 T	Brand 2x1–5·4,5–1. Q=10m^3/r, n=4 m. N$_{(эл.дв.)}$=4,5 kW, N$_{(эл.дв.)}$=970 min^{-1}
8	Parc	1	12 × 18 H10 T	L $_{ap}$=2140, D ap=273, P=6m^3
12,13,14	Containers for substances	5	Art. carbon	V=0,3m^3

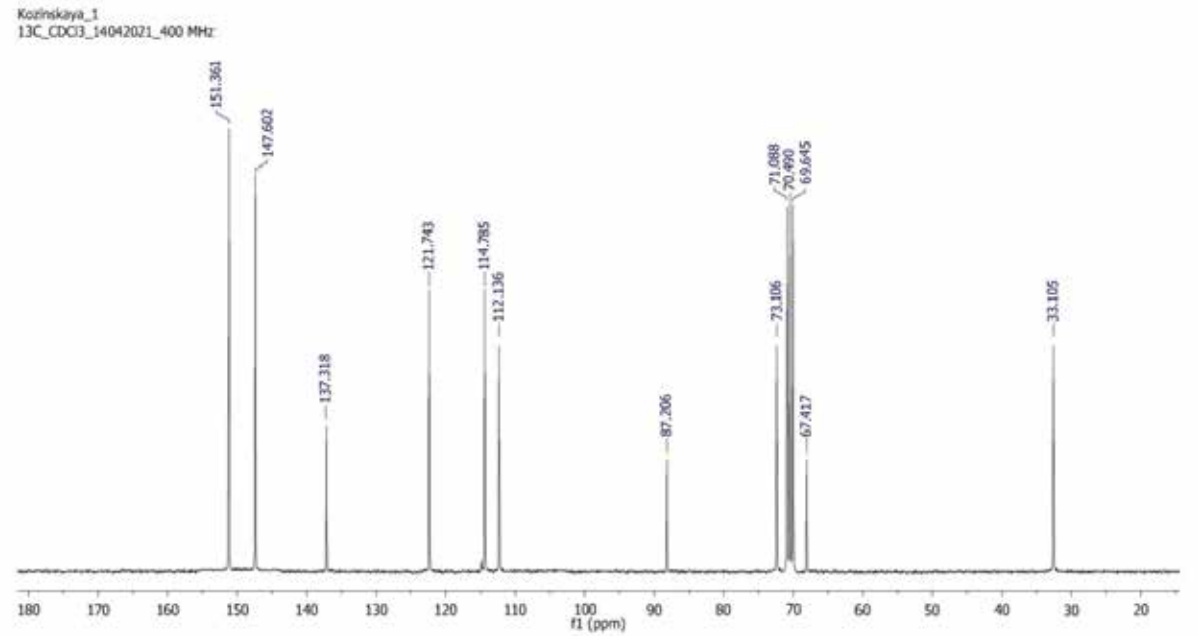
Where: V – apparatus capacity, m^3; Dapp – apparatus diameter, mm; H_{app} – apparatus height, mm; L_{app} – apparatus length, mm; P_{app} – pressure in the apparatus, MPa; P_j – pressure of the coolant, in the jacket, MPa; H – pressure, m.st.zh; N – motor power, kW; n – speed, min^{-1}; Q – pump capacity, m^3/hour

Table 11. *List of mandatory workshop instructions*

	Name of instructions
13.1.	Instructions for safety and fire safety of the workshop.
13.2.	Plan for eliminating emergency situations and accidents in the workshop.
13.3.	Instructions for preparation, delivery of equipment for repair and acceptance
13.4.	from repair
13.5.	Instructions for manual control of the fire extinguishing unit in workshops.
13.6.	Instructions for stopping the workshop for major repairs and starting the workshop after major repairs.
	Instructions for the synthesis operator of the installation for the production of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6
	Filter sheet for changes to the current regulations.



^{1}H-NMR spectrum of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6



^{13}C-NMR spectrum of 4′,4″-di-(1-methyl-1-hydroxyethynyl)-dibenzo-18-crown-6

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