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Kiev, USSR, 21 September - 1 October 1971
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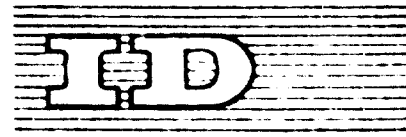
DEVELOPMENT OF NEW CATALYSTS FOR THE FIXATION OF
NITROGEN IN THE USSR^{1/}

by

A. M. Alexeyev

State Institute for Nitrogenous Fertilizers
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SUMMARY

DEVELOPMENT OF NEW CATALYSTS FOR THE FIXATION OF
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The development of modern technology for the production of ammonia, nitric acid, methyl alcohol, etc. in large-capacity plants has been made possible by a wide range of physico-chemical, technological, experimental and pilot projects concerned with research into the development and establishment of technology for the production of new catalysts for:

- the hydrogenation and absorption of sulphur compounds from natural gas;
- the steam and steam-air conversion of hydrocarbons;
- the low-temperature conversion of carbon monoxide;
- methanization;
- ammonia synthesis using a granular catalyst with reduction of the catalyst outside the column;
- the oxidation of ammonia with catalysts of reduced platinum content;
- the breakdown of nitrogen oxides;
- the breakdown of waste gas from nitric acid production, and the synthesis of methanol with a low-temperature catalyst.

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The development of new types of industrial catalysts is unthinkable today without a comprehensive study of **their** physico-chemical properties, and a wide variety of new physico-chemical methods of studying catalysts is therefore considered.

Modern technical progress in nitrogen industry as one of the leading branches of chemical industry is determined by the development of highly efficient large production lines based mainly on catalytic processes, and making use of a large number of highly reactive and selective catalysts.

Development of modern technology for the production of ammonia, nitric acid, methyl alcohol and such like products in large capacity reactors was made possible through carrying out physical-chemical, technological, experimental and semi-industrial research aimed at development and commercialization of new catalysts for hydrogenation and absorption of sulfurous compounds from natural gas, steam and steam-air hydrocarbon conversion, low temperature carbon monoxide conversion, methanation, ammonia oxidation with its lower platinoid content, nitrogen oxides decomposition, ^{decomposition} of exhaust gases from nitric acid production as well as at development and commercialization of ammonia synthesis granulated catalyst, including non-column catalyst reduction, and low temperature methanol synthesis catalyst.

Scientific research aimed at improving the catalysts produced by the industry were carried out in order to raise their activity, selectivity, stability, thermal resistance and mechanical strength and were based on comprehensive physical-chemical investigations of catalyst properties, better selection of raw materials and promoting additives, and on process and equipment improvement.

Extensive research carried out in Soviet nitrogen industry resulted in the development of new catalysts; basic technical data of these catalysts being cited below.

CATALYST FOR THE HYDROGENATION OF ORGANOSULFUR COMPOUNDS

The presence of organosulfur compounds, particularly, disulfides in the natural gas in the USSR necessitated the developing process of organosulfur compound hydrogenation to hydrogen disulfide in order to provide fine purification from sulfur. Nickel-molybdenum, molybdenum and cobalt catalysts on alumina support are mainly used in this process as contacts. New nickel-copper catalyst on alumina support has been developed in the Soviet nitrogen industry for the hydrogenation of organosulfur compounds. Propertywise this catalyst excels the other known catalysts (table N I).

Almost 100% hydrogenation with the volume velocity of 2000 -3000 hour⁻¹. $\left(\frac{\text{m}^3(\text{gas})}{\text{m}^3(\text{catalyst}) \text{ hour}} \right)$ at 250°-300°C is obtained through the utilization of a new catalyst.

Application of ^{the} new catalyst makes it possible to cut down the hydrogen amount for hydrogenation to 2-5%.

The catalyst manufacture technology is very simple, production cost of this catalyst is less than these of similar application. The catalyst is sufficiently strong, reliable in operation and can be successfully used for the purification of natural gas used in ammonia and methanol production.

CATALYST-ABSORBER FOR FINE PURIFICATION OF NATURAL GAS FROM SULFUR

Hydrogen disulfide formed as a result of hydrogenation of organosulfur compounds is usually absorbed on a zinc absorber. It should possess high specific surface and optimum porous structure. Only in this case can high purification degree be achieved, and the absorber has considerable sulfur capacity. A new, highly active absorber distinguished for its high strength and possessing high specific surface has been developed by the nitrogen industry of the USSR. In contrast to the other known absorbers this one absorbs practically total amount of hydrogen disulfide as well as all sulfurous compounds, contained in natural gas, thus making it possible to carry out purification from sulfur in one stage without hydrogenation. The catalyst has been tested on a semi-industrial plant.

Working conditions:

Pressure - 25-35 atm

Temperature - 280 -300°C

Volume velocity - 1000 -2000 hour⁻¹

Sulfur content in natural gas is cut down in this case from 2,0-14 mg/normal cubic meters to 0,0 - 0,3 mg/normal cubic meters (table 2). Purification system making use of the mentioned catalyst is distinguished for the equipment simplicity. The catalyst is available in the shape of 4x5mm tablets. Service life depends upon natural gas composition.

CATALYST FOR HYDROCARBON CONVERSION

New, original methods have been developed for the preparation of nickel containing catalysts for the conversion (reforming) of hydrocarbons mixed with various oxidants (water vapour, carbon dioxide, oxygen and air).

One of the catalysts obtained through the new method can be successfully used in the plants making nitrogen-hydrogen and hydrogen and hydrogen monoxide mixtures, pure hydrogen, inert gases and reducing gaseous mixtures at pressures up to 150 atm. By a large number of properties new catalysts excell considerably all known catalyst types used for the similar purposes (table 3).

Equilibrium of methane conversion reactions with the volume velocity on dry initial methane up to 4000 hour⁻¹ and initial mixture composition corresponding to the ratio of $\text{CH}_4:\text{H}_2\text{O} = 1:3$ can be practically obtained on one of ^{the} steam reforming catalysts working over 600-900°C temperature range at 30 atm pressure.

In contrast to the other known catalysts, activity of this catalyst practically does not change with its considerable overheating by 400-500°C over a long period of time in the course of conversion (for example, in the case ^{of} process temperature fluctuation

from 600 to 1100°).

The catalyst granules before as well as after use have high mechanical strength (compression resistance - up to 1000 kg/cm²), exceeding the strength of known analogous catalysts.

Thermal stability of this catalyst is 2-3 times higher than that of known catalysts. New catalyst stands more than 50 sharp heat changes with temperature fluctuations from 20 to 1200°C. without signs of destruction.

The catalyst preparation method is distinguished for its simple technology and few number of stages. Complete absence of solid, liquid or gaseous harmful wastes is a considerable advantage of the new technology.

Another type of catalyst also containing small amount of silicon dioxide has its mechanical strength, in the course of operation, reduced only by 15-30% and not by 70-90% in contrast to known catalysts made by foreign companies (C&I Girdler - G-56; ICI -57 - 1, CCI - C - 11 - 2 S).

Particles of new catalysts can be made in any shapes, for example, cylinder, ring and ball.

CATALYST FOR CARBON MONOXIDE CONVERSION

New, highly efficient carbon monoxide conversion catalyst have been developed by the USSR nitrogen industry as a result of the implementation of a wide research program.

6.

It favourably differs from the known catalysts by its higher activity over a wide temperature range (180-500°C), good mechanical strength (the ^{tablets} can stand crushing force up to 40⁰ kg/cm²) and simple technology of its manufacture (table 4).

High thermal stability (the catalyst shows stable performance at temperatures up to 500°C), and high activity makes it possible to utilize this new catalyst successfully for the 1-st and 2-nd stages of carbon monoxide conversion.

The use of new catalyst results in considerable increase of hydrogen monoxide conversion plant efficiency due to lower steam/gas ratio, higher volume velocity and longer catalyst service life. Besides, catalyst utilisation in both stages makes unnecessary filling with sulfur removal substance before low temperature catalyst and desulfurization (required when using iron-chromium catalyst).

The catalyst is available in tablets or granules of different shapes and sizes.

METHANATION CATALYST

Laboratory research and studies in semi-industrial conditions have resulted in the development of a new highly efficient nickel catalyst on a stable heat resistant support, to be used in the plants for the purification of nitrogen-hydrogen mixture and hydrogen from carbon oxides and oxygen, through their hydrogenation into methane and water.

Property-wise this catalyst excels many known catalysts used for the same purpose (table N5).

7.

This catalyst has high activity, heat-resistance and mechanical strength. Operating pressures are 10-500 atm, temperatures = 250-350°C. Its volume speed ranges from 2000 to 200 000 hour⁻¹. It remains active when overheated to 600°C.

Through the utilization of the new catalyst carbon oxide content in nitrogen-hydrogen mixture and in hydrogen can go down to 5-10 cm²/m³. Besides, this catalyst can be used for the hydrogenation of some organic substances. The catalyst is available in 6x6 mm tablets.

AMMONIA SYNTHESIS CATALYST

Catalyst CA-1 (State Standard 12411-66) is used in ammonia synthesis columns in the Soviet nitrogen industry.

By its catalytic properties (activity, stability, mechanical, strength) this catalyst stays on the same level with the best foreign catalysts. (Table 6). Considerable changes having occurred within the last years in technology of the catalyst manufacture made possible production of this catalyst in granulated and pre-reduced form, thus permitting to improve the catalyst quality and in doing so to intensify ammonia synthesis process.

The granulated catalyst is distinguished for higher mechanical strength and better abrasion resistance in contrast to the ground catalyst. Utilization of granulated catalyst (as indicated by industrial test results) in ammonia synthesis columns with diameters ranging from 700 to 1400 mm makes it possible to cut down hydraulic resistance of the plant, raise its output by 10-150% through the use of fine grains, and to decrease the apparatus erosion. Due to lesser build-up and carryover of catalyst dust, iron content in ammonia is reduced by the factor

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of 1,5; clogging of heat exchange surfaces with the dust goes down, thus increasing efficient utilization of ammonia synthesis reaction heat.

The catalyst is available in regular spherical granules with 4-5, 5-7, 7-10 mm diameter.

The catalyst service life is guaranteed for 3 years.

Non-column reduction of ammonia synthesis catalyst has been commercialized. In the case of non-column reduction, optimum introduction of the contact formation process is made possible, thus permitting to raise its activity. The column start-up and commissioning time can be saved by the factor of 3-4.

In the nearest future all the nitrogen industry plants in the USSR will change over to the utilization of reduced granulated ammonia synthesis catalyst.

AMMONIA OXIDATION CATALYST WITH A LOWER PLATINOID CONTENT.

The USSR is one of the largest manufacturers of nitric acid. Special alloy screens with 90-95% platinum and 5-10% rhodium content are used in many countries as ammonia oxidation catalysts, whereas in the USSR and in some other countries these screens are made from the alloy with 92,5% platinum, 3,5% rhodium and 4% palladium content.

Prolonged scientific research in the Soviet Union has resulted in the development of a new catalyst for ammonia oxidation into nitrogen oxide.

Thorough and comprehensive catalyst tests, prolonged experience of its industrial application has shown that the new catalyst is not inferior, by its basic properties, to the conventional catalysts, used for ammonia oxidation.

Ammonia conversion degree on this catalyst reaches 97-98,5% at atmospheric pressure, whereas at 6 atm. abs. this figure goes down to 94-96%.

The screens are made of 0,09 mm diameter wire with 1024 bradings per 1 cm square. The new catalyst is favourably distinguished from the other catalysts, used at present, by the following features:

- Certain amount of platinum in the catalyst composition has been substituted by less expensive metals.
- The weight of catalyst screens is less by 8-10% per surface unit, thus providing corresponding decrease in specific, initial catalyst consumption in the course of nitrogen oxides and nitric acid production..
- With the use of the new catalyst specific irreplaceable losses of precious metal in the course of ammonia oxidation go down by 15-25% and amount approximately to 0.035 gr. of platinum per ton of nitric acid at atmospheric pressure and to 0.12 gr of platinum at 6 atm. abs.
- The catalyst is less sensitive to catalytic poisoning.
- The service life of these screens is 12-14 months for the installations working at atmospheric pressure and 6 months for the installations working at 6 atm. abs.

Less operation losses in the manufacture of nitric acid and nitrogen oxides are made possible through the application of new catalyst.

The new catalyst is suitable for use in any ammonia oxidation systems with the pressure of 6 atm. abs.; it can also be used in operating systems of nitric acid and nitrogen oxides production without alteration of existing contact apparatuses.

Used up new alloy catalyst screens can be regenerated in the same systems where conventional platinum-rhodium screens are regenerated.

New equipment is not required for the catalyst manufacture.

Double stage ammonia oxidation catalyst containing non-platinum catalyst in the second stage, made from cheap and available raw material, is used in the nitrogen industry of the Soviet Union. Its service life is not less than 5 years. The use of double, stage catalyst makes it possible to cut down precious metal consumption by 50% and irreparable losses by 20%.

CATALYST FOR THE DECOMPOSITION OF NITROGEN OXIDES CONTAINED IN EXHAUST GASES .

Double layer catalyst for the purification of exhaust nitrose gases through high temperature decomposition of nitrogen oxides has been developed and successfully used in nitrogen industry of the USSR. Catalytic decomposition is carried out through the use of various reducing agents (hydrogen, tank and purge gases, methane from natural gas).

Nitrogen oxides content in purified exhaust gas is 0.005 vol.pct. with the outlet gas temperature of 700-750°C and pressure of 6 atm. Operation life of the catalyst is 1.5 year. At present, medium temperature catalyst for the purification of exhaust nitrose gases from nitrogen oxide, using ammonia as reducing agent, is being semi-industrially tested.

When developing industrial types of catalysts great attention during research was drawn to studying their chemical-physical properties, and, therefore, we shall specially dwell on this topic with the view of possible discussion.

STUDIES OF PHYSICAL-CHEMICAL PROPERTIES OF CATALYSTS

At the present time, development of industrial catalysts almost always necessitates complex study of their physical-chemical properties. The fuller physical-chemical parameters of solid contact are given, the faster the selection of the industrial catalyst optimum chemical composition, conditions of its manufacture and utilization is made.

For a long time the methods for the determination of the total surface, the catalyst porosity, radial pores distribution as well as x-ray graphical analysis methods for the determination of the phase composition of the samples and their dispersion degree and the methods for studying mechanical strength were considered as the basic methods for the determination of industrial catalyst properties. However, industrial application experience points out that the above mentioned methods are obviously insufficient.

Insufficiency of existing methods consists mainly in that they all allow to analyse the catalyst in its final state, not in the course of its processing; it should be also noted that the catalyst can, as a rule, be analysed at room temperature, and in the open air. Therefore, the mechanism and dynamics of phase, chemical and other transformations, occurring in the catalyst during its processing in an industrial reactor remain, to a considerable extent, not yet revealed. In this connection, ^{at present} the problem of introducing new methods into the practice of physical-chemical studies of industrial catalysts has become particularly acute, the main requirement being the possibility to carry out various analyses under ^{approximating} conditions, ~~resembling~~ industrial ones (high temperatures, elevated pressures, flow conditions, utilization of gas mixtures etc.)

It is a common knowledge that the most modern physical chemical methods are used, preferably, to study "pure" systems leaving aside industrial catalysts due to their complicated chemical composition. Besides, a large number of research workers concentrated mainly on studying catalytic reaction mechanism and to a lesser extent on studying formation mechanism of industrial catalysts at various stages of their manufacture (manufacture and heat treatment of contact substance, heating, reduction etc.)

Knowledge of interaction mechanism between separate catalyst components in a solid phase and effect of various gases, temperature, pressure etc. upon this interaction character is essential for the selection and development of active, heat resistant and stable in operation industrial catalysts.

This trend has enjoyed a large growth as applied to the investigations of commercial catalysts used in nitrogen industry. At present, differential thermal analysis combined with mass-spectrum and chromatographic analysis of gaseous products, as well as differential thermal analysis together with weight change recording, thermo-magnetic analysis, infrared spectroscopy and x-ray structural ~~analysis~~ ^{analysis} of phase composition of catalysts are used as the main methods for studying catalyst formation processes. Electron diffraction and electron microscopy methods are used to study the catalyst surface microstructure. Local x-ray spectral analysis method is used for the determination of small concentrations of chemical elements in a catalyst and distribution of the elements of the basic components and micro-additives along the phases.

Thermographic, chromatographic mass-spectrum and thermo-magnetic analyses of catalysts are carried out with the samples ^{being} heated in the flow of various gases. A unit has been commissioned carrying out differential-thermal analysis of the processes taking place in solid phase at pressures up to 100 atm. in static as well as in dynamic states. Complex utilization of the above methods makes it possible to obtain extensive information concerning nature and stages of chemical and phase transformations in a catalyst and its components in the course of heat treatment, reduction as well as in the course of the catalyst poisoning and deactivation.

Besides, the application of thermographic analysis makes it possible to assess the character of heat changes in an industrial reactor with the catalyst being properly treated, thus presenting great practical interest for the selection of reactor design and for developing the process of catalyst manufacture.

Thus, for example, reduction of various nickel-chromium

catalysts based on hexavalent chromium compounds is attended with considerable exothermic effect (400° / 1 gr of catalyst) recorded by differential thermal analysis. Temperature range of this effect ($190-330^{\circ}$) and the nature of the exothermal process observed have been established through detailed physical-chemical investigations.

Activation of the industrial nickel-chromium catalyst is attended with an exothermal process over the temperature range of $120-200^{\circ}\text{C}$ (fig.2) which is governed by the nature of the support. Similar effect is not detected for nickel-aluminium catalyst with the same nickel content and analogous method of manufacture.

Application of mass-spectrum method has extended the possibility of gas phase analysis. In the case of continuous control this method will make possible sufficiently complete identification of gaseous phase composition and change of its components, including reactive and short-lived compounds in the course of the catalyst heat treatment in vacuum and in various gas media.

The presence of ions with the mass number m/e 18.44; 32.16 has been established by means of mass-spectrum analysis in the course of contact mass heat treatment up to 600°C in vacuum, thus indicating liberation of H_2O , CO_2 , O_2 , O and such like substance into the gas phase.

The results obtained through the application of the above methods as well as differential thermal analysis data made it possible to explain a number of chemical transformations occurring in a solid phase in the course of contact mass sample roasting.

Application of local x-ray spectral analysis method for industrial catalysts is a new trend in the field of heterogenous catalysis studies in the Soviet Union as well as abroad.

Local x-ray spectral analysis is, so far, the only direct method for the observation of the distribution of the elements along the phases in the region of a few microns. This method combined with petrographic analysis is used, in the case of an industrial catalyst, for the control of mineralogical phases composition and elements redistribution along the phase depending upon the ratio of initial components, method of the catalyst manufacture, its heat treatment and reduction conditions as well as in the course of the catalyst deactivation (overheating, copper and water vapour poisoning).

Thus, it has been established that potassium and aluminium are distributed non-uniformly along the phases in the oxidized and twice-promoted iron catalyst ($\text{Fe-Al}_2\text{O}_3\text{-K}_2\text{O}$). Potassium, after having been reduced, is concentrated in iron containing phases and distributed uniformly in α -Fe. After overheating the catalyst over to 500°C aluminium is coagulated on the boundaries of grains α -Fe, whereas potassium leaves grains α -Fe completely.

The whole complex of physical-chemical studies was successfully made use of in the course of the development of industrial methanation catalysts, when studying formation processes of low temperature carbon monoxide conversion catalyst and in the course of studying the processes of ammonia synthesis catalyst deactivation.

Implementation of an extensive research program aimed at the development of new catalysts for the manufacture of fixed nitrogen in the USSR, made it possible to develop various types of catalysts meeting specifications for the best foreign catalysts and by certain properties exceeding them.

By now, the Soviet nitrogen industry has accumulated sufficient experience in the development, designing and setting up capacities for the manufacture of new catalysts and can engineer separate plants for making individual catalysts as well as large catalyst factories making a wide range of these products.

TABLES AND FIGURES

TABLE N 1

Comparative data on alumocobaltmolybdenum catalysts

(with the volume velocity of 2000 hour⁻¹
and ^{at}atmospheric pressure)

Type of sample	Temperature °C	Hydrogen content %	Sulfur content mg/nm ²		Degree of hydrogenation %
			Before test	After test	
USSR (industrial)	350	5	26.0	0.4	98.5
BASF (Bundesrepublik)	350	5	22.9	0.7	96.1
C&I Girdler (USA)	350	5	19.9	1.0	95.0
Grande Paroisse (France)	350	5	31.2	0.3	99.0
COI (U S A)	350	5	24.6	2.1	91.5

TABLE N 2

Comparative data on zinc^{oxide} based catalysts

Type of catalyst	Shape and size of particles	Bulk weight kg/l	Porosity	Specific surface m ² /g	Capacity for sulfur % of the weight at 400°C
USSR (industrial)	Tablets diam.-5mm height-3 mm	1.57	55.0	32.8	24.5

USSR (semi-industrial)	Tablets diam.-5 mm height-3mm	2.11	36.7	45.0	29.4 (t=250°C)
32-4 (ICI, U.K.)	Balls diam.-3mm	1.19	43.3	34.9	20.2
29-2 (ICI; UK)	Tablets 5x5	1.93	30.3	35.7	24.3 (t=250°C)
B-ZnO (BASF, Bundesrepublik)	Molded diam.-4mm height-5-10mm	1.44	44.8	18.7	26.3
741 (NIKKI, Japan)	Tablets diam.-5mm height-4mm	1.07	55.3	29.2	30.2
ZnO (ONIA, France)	Tablets diam.-10mm height-5mm	1.55	39.5	35.6	18.0
O-72-C (O.I Girdler, U S A)	Balls diam.-3mm	1.23	58.6	-	21.4

TABLE N 3

Specification of hydrocarbon conversion catalyst with low silicon content

Type of catalyst	Shape and size of tablets mm	Temporary resistance to compression		Strength losses	Thermal resistance (number of thermal cycles: 20x1000°C -20°C in air, without deactivation)
		initial kg/cm ²	after conversion on kg/cm ²		
USSR (industrial)	Cylinder 12x12	1500	1500	-	30
USSR (semi-industrial)	Rings diam-12x6 height-12	1000	940	10	40
	Cylinder 12x12	1500	1350	10	50
USSR (semi-industrial)	diam-14x7 height-13	630	440	30	30

TABLE N4

Comparative data on low temperature CO
conversion catalysts

Type of catalyst	Residual CO at various temperatures, %				Maximum temperature not causing decrease in activity
	150	175	200	225	
LFC (USSR)	0.8	0.24	0.0	0.1	up to 500
BTT (BASF, Bundesrepublik)	4.4	1.2	0.3	0.13	350
G-66B (C&I Girdler U S A)	8.0	2.1	0.55	0.55	450
GP-C6 (Grande Paroisse France)	12.1	8.0	1.7	0.7	300
ONIA (France)	14.8	14.6	13.2	9.8	300
G-18 (CCI, USA)	7.0	1.2	0.25	0,16	400

Gas: H₂ - 53%

CO - 19%

CO₂ - 8%N₂ - 5%Volume velocity - 1000 hour⁻¹

steam/gas ratio - 1,5

Pressure - atmospheric

TABLE N5

Comparative data on methanation catalysts

Type of catalyst	Activity ^x °C	Thermal stability °C	Mechanical crushing strength kg/tablet
USSR (semi-industrial)	140	600	25
BASF, Bundesrepublik	180	500	6
Q.I Girdler USA (G-65)	170	650	25
OCI (C-13-4), USA	160	450-480	9
ONIA, France	180	550	10
P.K., Denmark	200	500	6

x/ temperature of carbon oxide breakthrough
at 15 atm.pressure
Velocity - 4000 hour⁻¹

TABLE 26

Comparative data on ammonia synthesis
catalysts

Ammonia content in vol. pct. in an exhaust gas
at 300 atm. pressure with volume velocity
of 30 000 hour⁻¹

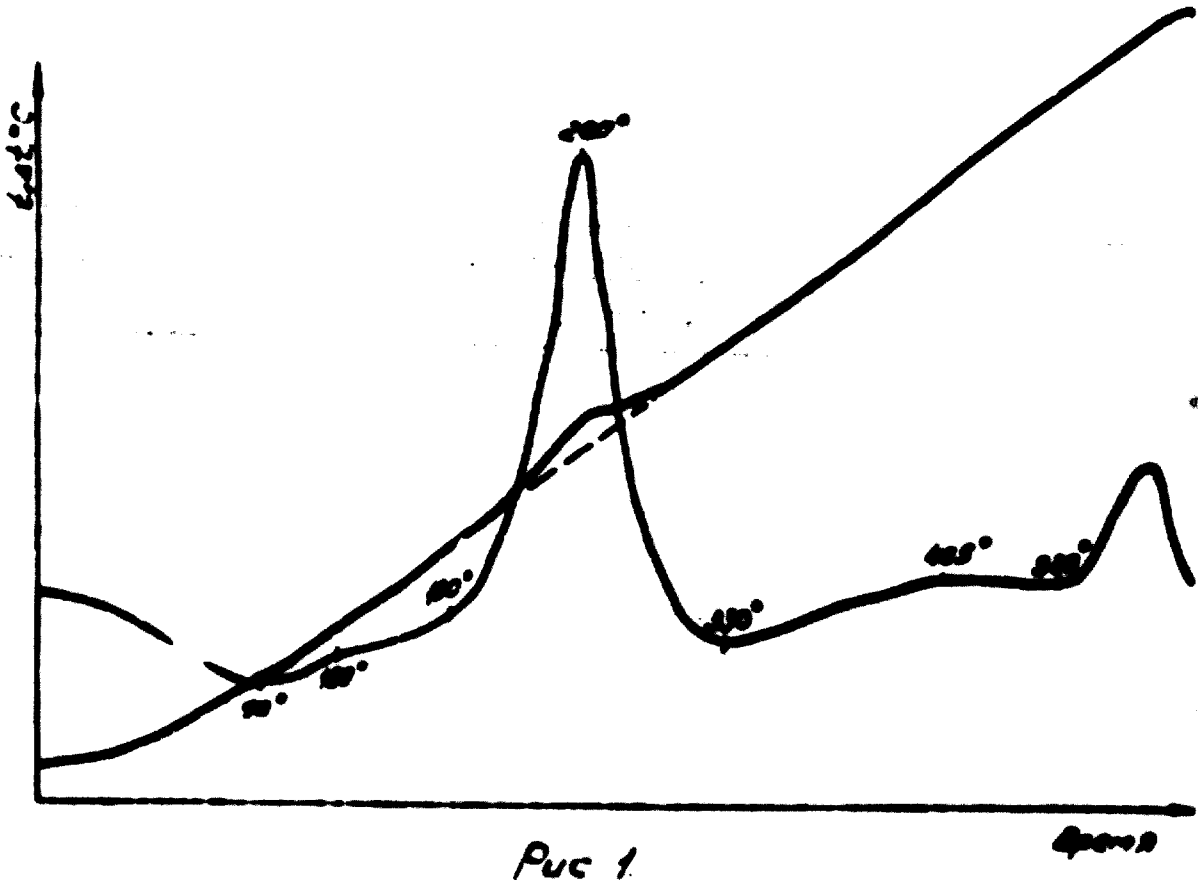
Type of catalyst	Temperature °C				
	400	450	475	500	550
SA-1 (USSR)	17.7	23.6	24.4	23.7	17.0
BASF (Bundesrepublik)	17.0	23.0	23.8	21.2	16.4
EM-7 (Sweden)	16.6	20.3	20.4	19.6	15.2
35-4, ICI (England)	17.9	20.5	-	19.5	15.5

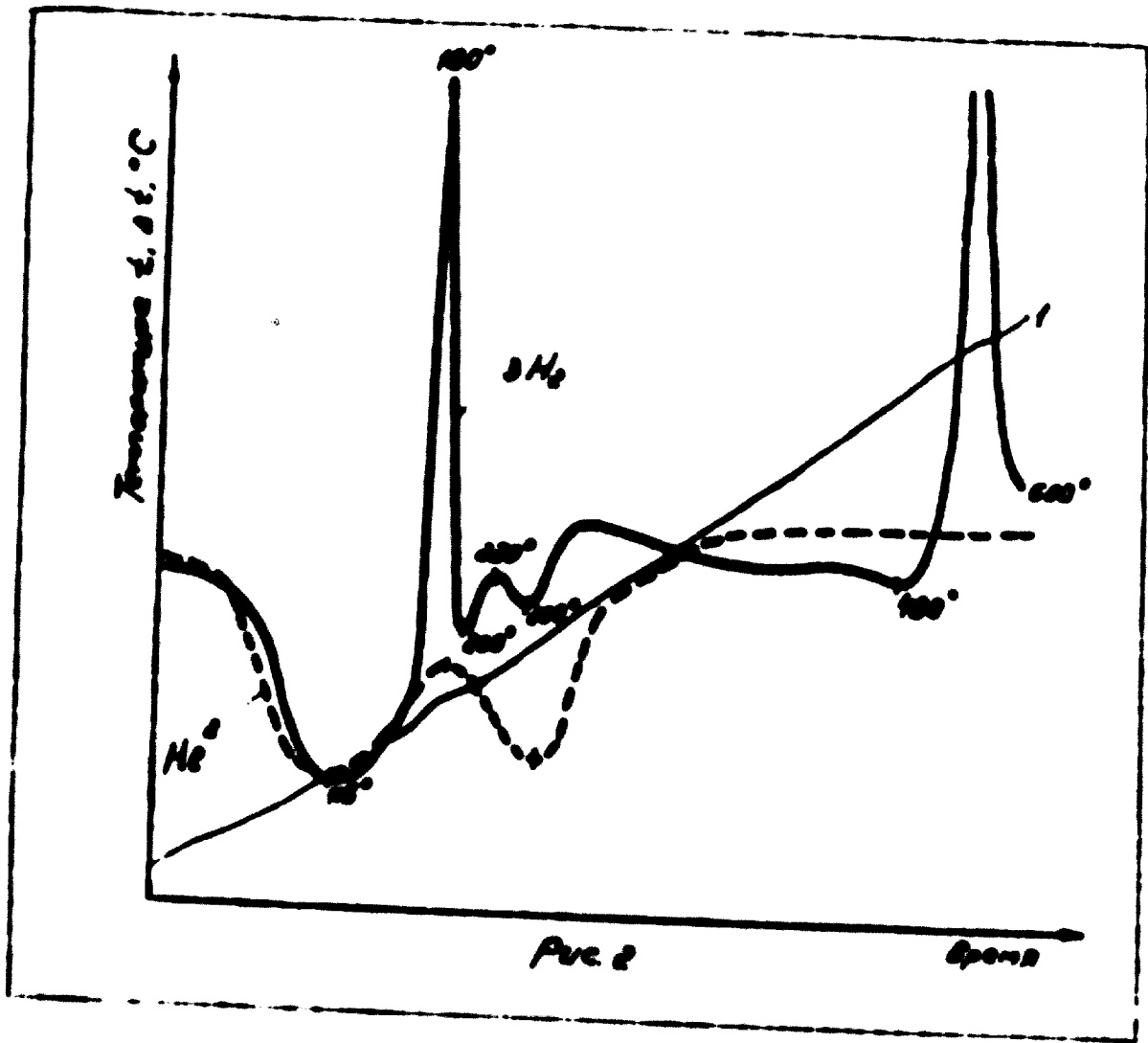
Captions

Fig. 1 Reduction of nickelchromium catalyst with hydrogen

Fig.2. Differential thermal analysis of the activation process
of an industrial
nickelchromium catalyst with hydrogen

Fig.3 Mass-spectrum analysis of thermal decomposition products
of carbon monoxide catalyst.





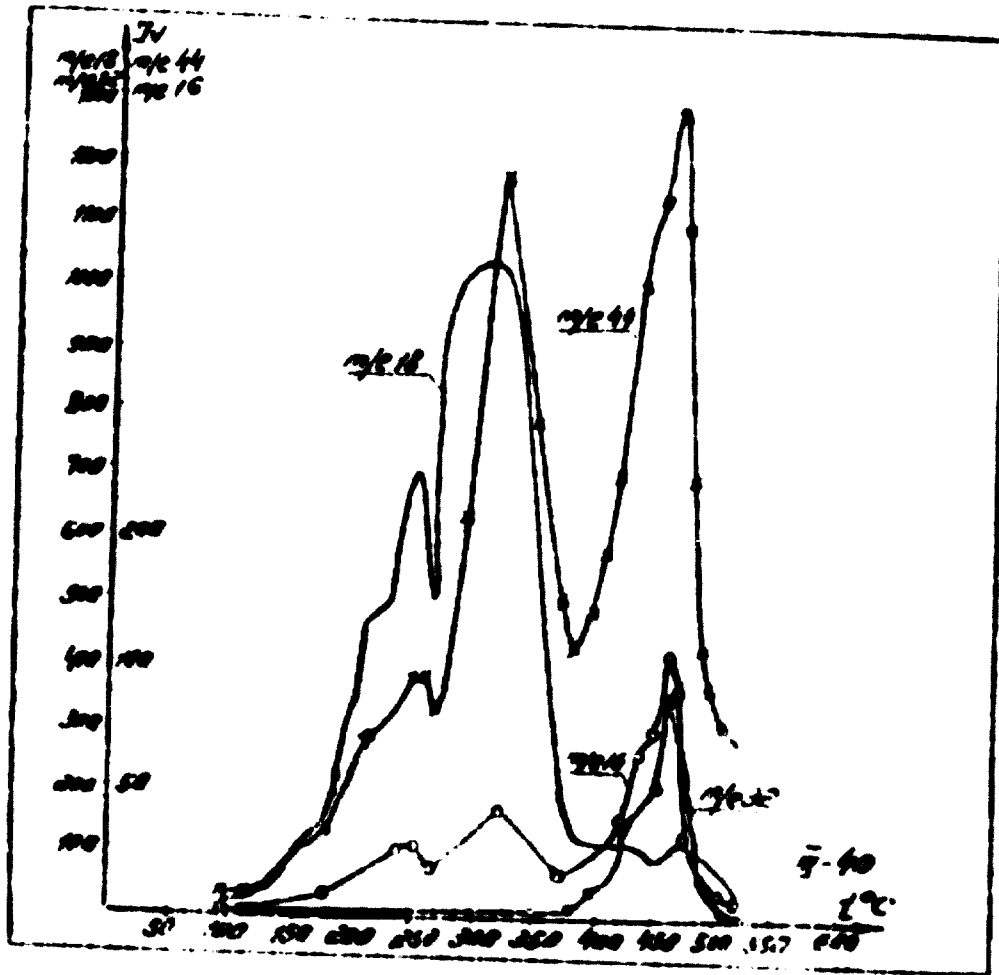
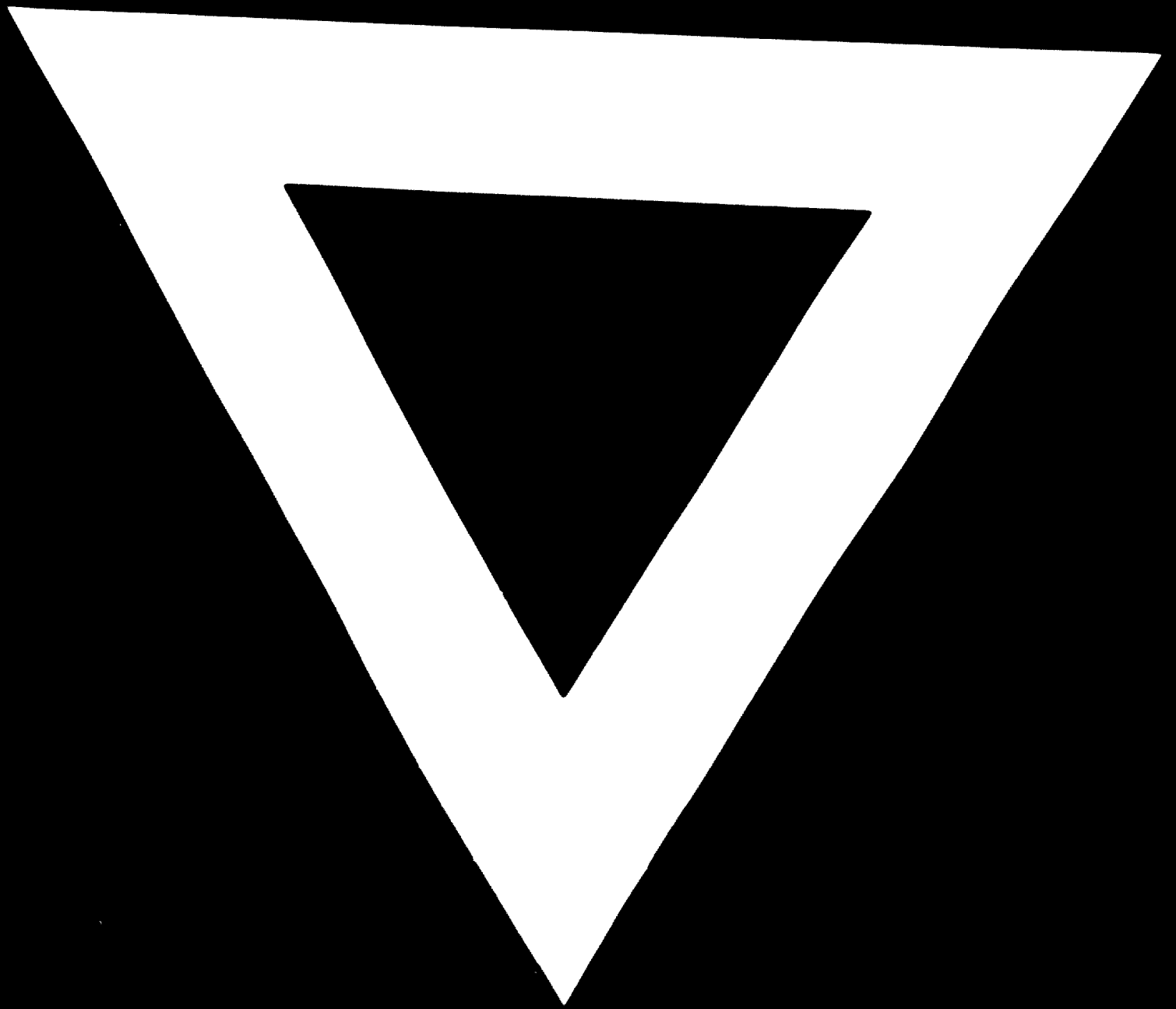


Fig. 3.





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