Research on Environmental Issues of Ammunitions Recycling and Development of Catalytic Method for Purifying Emissions Caused by their Thermal Utilization Process

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Abstract

Analysis of environmental issues linked to the thermal treatment of ammunition was carried out. An opportunity to purify ventilation emissions from thermal treatment furnace using a sorbent and deep oxidation catalyst was explored. The activity of a series of complete oxidation catalysts based on transition metal oxides, and also platinum and palladium was investigated. It was determined that Pt–Pd/5 % CeO_2–Al_2O_3 catalyst displayed the highest activity. A technology was suggested and a plant to dispose of ventilation emissions from the thermal utilization of sub-standard ammunition (cartridges, casings, and small arms primers) was developed. The technology includes removal of vapours of mercury, lead, nitrogen oxides, and other inorganic impurities from the gases on a sorbent and CO oxidation over a solid catalyst.

Key words: pyroxylin powders, initiating explosives, gas treatment, sorbent, catalyst, catalytic oxidation

INTRODUCTION

A great deal of sub-standard ammunition has been accumulated worldwide, the environmentally safe recycling of which poses a big problem. For example, significant amounts of ammunition have been accumulated at arsenals, bases, and storages of the Ministry of Defence of the Russian Federation. The former in the mass equivalent reaches dozens of millions of tons. Various ammunition has guaranty periods of storage or has already been withdrawn from service [1]. When recycling explosive materials (EM), powders that initiate them, tracers, highly toxic compounds are evolved. The latter involve vapours and aerosols of heavy metals, such as lead and mercury, antimony trioxide and barium oxide, and also toxic gases, *i.e.*, nitrogen oxides and sul-

phur oxides, and also carbon monoxide. Herewith, there is atmospheric pollution with these toxic compounds upon both ammunition disposal and thermal neutralization [2, 3].

Disposal methods for smokeless powders and powder-containing products are based on chemical or thermal recycling. There are numerous patents related to the chemical recycling of powders to useful products. Thus,

Russian patent [4] describes the recycling of smokeless powders by autoclave polymerization into materials used as nitrocellulose paints and varnishes, and also household products instead of mastic-lacquer colloxylins. A preparation method for cellulose epoxinitrates based on powder waste for the polymer frame of hard polymeric coatings, clays, and solid fuels is suggested in [5]. The patent [6] reports powder chemical processing

into cellulose acetonitrate compounds used in formulations of etrols, industrial explosives, and also mastic-lacquer and other compounds.

Chemical and thermal methods are combined in [7]. The suggested method assumes the chemical treatment of powder waste with aqueous solutions of salts and clay additives with a view to producing a compound with a long time of combustion. That enables to eliminate an opportunity for detonation (explosion) upon bakeout and control the combustion process of waste powders and therefore meets the requirements for the conduction of a safe disposal procedure for powders. The patent [8] proposes a similar method for recycling, whereat water and an inert material are added to the powder, whereas the combustion process of the resulting composite is carried out safely in the fluidized bed of a nonreactive substance. A device (an armored incinerator) for the thermal treatment of ammunition is described in [9]. Toxic exhaust gases are let through a system of filters neutralizing them till a safe concentration. The patent [10] reports a fluidized bed combustion plant. Explosive material residues are burnt in a mixture with waste water that contains organic matter to guarantee process safety.

A system for the thermal utilization of a powder residue on shell casings was suggested in a PCT application [11]. Gases with toxic impurities formed resulting from the treatment were treated in a dust collector with a filter and then in a post-combustion chamber.

As shown by a review of patent literature, only a few patents suggested thermal utilization installations with a system for waste gas treatment. Generally, gases are discarded without treatment or purified only of solid impurities on filters. No data has been found for the use of catalysts for gas treatment in the patent literature.

Similar results were also acquired upon scientific and technical literature analysis. There is a shortage of research works describing gas treatment systems after the thermal treatment of ammunition. For example, the treatment system of gases caused by thermal disposal of thermal recycling of ammunition is described in [2] and involves a cyclone, a particulate trap, and an absorber for wet gas treatment. However, this system does not envisage the use of catalysts.

Toxic gas emissions are generated in special burners upon the thermal utilization of defective ammunition, in particular, cartridges and casings of primers. The emissions may contain vapours of mercury and lead, Sb_2O_3, nitrogen oxides, sulphur compounds (H_2S and SO_2), and carbon monoxide (CO) [12, 13]. As demonstrated above, the methods used to purify these emissions are not efficient enough. At the same time, ever toughening requirements towards environmental protection dictate the need for using economically viable purification technologies for gas emissions generated upon ammunition recycling.

Promising purification methods for gas emissions are based on using catalysts. The major components of gas treatment systems are catalytic reactors that are capacities with the required amount of the catalyst. Emissions purification till statutorily prescribed standards is performed therein. Purification processes are grounded on complete oxidation of organic impurities and CO in emissions over solid catalysts to form CO_2 and H_2O. Nitrogen oxides and sulphur oxides, and also mercury vapours may be removed out of the emissions using sorbents.

Relying on the data of [14–38], and also on the experience on producing catalysts for complete oxidation at the Boreskov Institute of Catalysis, Siberian Branch, Russian Academy of Sciences (Novosibirsk, Russia), one should expect the high activity of catalysts based on noble metals (Pt and Pd) in oxidation reactions of hydrocarbons and carbon monoxide. Selecting a specific catalyst for practical use should be based on the investigation of catalytic activity.

The objective of the research was to investigate an opportunity to purify furnace ventilation emissions from the furnace used for the treatment of sub-standard small arms cartridges using sorption and catalytic methods.

EXPERIMENTAL

Waste gas composition

The utilization of sub-standard products and production waste, *i.e.* cartridges, casings with percussion caps, and the latter alone is conducted at one of the enterprises for ammunition production in an electric furnace with an inner screw. The furnace temperature is 500–750 °C. The products are fed to the screw. When the latter is rotated, the products enter the hot area of the furnace where the powder, pyrotechnic formulations, and initiating explosives are thermally decomposed. At the outlet, the treated products enter the discharge pan, above which there is a ventilation hood. The waste gas of the furnace is diluted with air, sucked off using a ventilator, and discarded into the atmosphere. A mechanical FBP 70–40 filter is currently envisaged for gas treatment.

Analysis of ventilation emissions of the furnace was performed using of a Polar gas analyser manufactured by "Promecopribor" Ltd. (Russia) upon the thermal treatment of demilled 7.62 mm cartridges (with minimum powder residues). As demonstrated by measurements, the major component of emissions is CO (a concentration of $0-200$ mg/m^3 therein, an average content of around 100 mg/m^3); NO, NO$_2$, and H$_2$S may also be present in small amounts.

Preparation of catalyst laboratory samples

In order to prepare catalyst laboratory samples, the method of incipient wetness impregnation with aqueous solutions of salts precursors of the active component with a given concentration was used.

With a view of preparing laboratory samples of Mn−La/AlSi catalyst, a ceramic carrier based on α-Al$_2$O$_3$ and α-SiO$_2$ (0.5−1.0 mm fraction) and an aqueous solution of a mixture of manganese nitrate and lanthanum nitrate was used. The catalyst calcination temperature and duration were 550 °C and 4 h, respectively. The total content of oxides in the catalyst was 5 mass %.

Samples of 20 mass % CuCr$_2$O$_6$/Al$_2$O$_3$ catalyst were prepared by incipient wetness impregnation of spherical γ-alumina pellets (1.0−1.6 mm) with a specific surface area of 193 m^2/g by an aqueous solution of copper dichromate for 15 min. Afterwards, the impregnated pellets were dried under an IR lamp for 1 h and air calcined at 700 °C for 4 h.

A similar method was used to prepare 20 mass % Cu$_x$Mg$_{1-x}$Cr$_2$O$_4$/Al$_2$O$_3$ catalyst. However, an impregnating solution of copper dichromate and magnesium dichromate in the required proportion were employed.

Samples of Pt − Pd/5 % CeO$_2$−Al$_2$O$_3$ catalyst were prepared by incipient wetness impregnation of 5 % CeO$_2$−Al$_2$O$_3$ support with a solution of tetrachloropalladium and hexachloroplatinum acids used in the required quantity. After impregnation, the resulting pellets were dried at 120 °C and calcined at 550 °C for 1 h and 4 h, respectively.

Catalyst physicochemical investigations

Elemental analysis was carried out by X-ray fluorescence using an ARL-Advant's analyser with Rh-anode of the X-ray tube.

Texture characteristics, such as specific surface area (A_{BET}) and pore volume (V_{pore}), were explored using an ASAP-2400 Micrometrics automated volumetric plant by low-temperature nitrogen adsorption at 77 K. The value of specific surface area was calculated according to the BET equation.

The mechanical strength for crushing was determined with an MP-9C instrument. The essence of the method consisted in measuring the effort required to destroy the sample (pellets and rings) among two parallel planes. The value of strength along generatrix (P_g, kg/cm^2) was calculated by the formula:

$$P_g = p/S = p/(dL)$$

where p, S, d, and L are the indicator reading of the instrument (kg), sectional area (cm^2), diameter (cm), and height (cm) of the sample, respectively. The difference between the diameter and sizes of inner port holes was accepted as the ring diameter (d).

The most active catalysts for oxidation of hydrocarbons and CO were selected for research. These included manganese-lanthanum (Mn−Al/Al−Si), copper-magnesium-chromium (20 %

TABLE 1

Physicochemical properties of the catalysts

Catalyst	Appearance	Content of active component, wt. %	A_{BET}, m^2/g	Pore volume, cm^3/g	Mechanical strength along generatrix, kg/cm^2
Mn−La/AlSi	fraction 0.5−1.0 mm	Mn 2.19 La 1.37	18	0,20	−
20 % Cu$_x$Mg$_{1-x}$Cr$_2$O$_4$/Al$_2$O$_3$ $x = 0,2$	Balls, diameter 1.0−1.6 mm	Mg 1.41 Cr 10.4 Cu 0.96 Fe 0.03	110	0,34	281
20 % CuCr$_2$O$_4$/Al$_2$O$_3$	Balls, diameter 1.0−1.6 mm	Cu 5.36 Cr 8.71 Mg 0.04 Fe 0.03	125	0,34	378
Pt−Pd/5 % CeO$_2$−Al$_2$O$_3$	Rings, diameter 8 mm, height 7 mm, hole 2 мм	Pt 0.18 Pd 0.10	89	0,43	29

$Cu_xMg_{1-x}Cr_2O_4/Al_2O_3$, $x = 0.1-0.20$, copper-chromium (20% $CuCr_2O_4/Al_2O_3$), and platinum-palladium ($Pt-Pd/CeO_2/Al_2O_3$).

Table 1 reports characteristics of the chemical composition, specific surface area (A_{BET}, m^2/g), and mechanical crushing strength of the g catalysts.

Method for measuring catalytic activity

The catalytic activity of laboratory samples was explored in the model reaction of complete oxidation of CO using a laboratory-scale setup (Fig. 1) with a flow type reactor at temperatures between 50 and 250 °C. The sample was loaded into the reactor. The initial reaction mixture containing 1 vol. % of CO in air was fed into the reactor at a gas hourly space velocity (GHSV) of 1000, 4000, and 10 000 h^{-1}. Catalytic activity was evaluated according to the temperature of 50 % conversion of CO. Analysis of the reaction mixture before and after the reactor was carried out using LKhM-8MD gas chromatograph with a thermal conductivity detector. Helium was used as a carrier gas. Operating columns are NaX and Povorak T. A detector was catarometer, $I_{det} = 150$ mA, the temperatures of the detector with the evaporator and the column were 150 °C and 28 °C, respectively.

RESULTS AND DISCUSSION

Catalyst selection

An experimental investigation on the effect of the chemical nature of the active component on

catalytic activity in the CO oxidation reaction was performed. As ascertained, the chemical nature of the active component has an effect on the catalytic activity of samples.

Figure 2 presents CO conversion *vs* catalyst composition relationships. As shown, catalysts based on noble metals, $Pt-Pd/5\%$ $CeO_2-Al_2O_3$ (curve *4*) and $CuCr_2O_4/20\%$ $CuCr_2O_4/Al_2O_3$ (curve *3*), display high catalytic activity in the CO oxidation reaction. The temperature of 50 % CO conversion ($T_{50\%\ CO}$) is 140 °C. The combined introduction of magnesium dichromate and copper dichromate results in a reduction of the catalytic activity of samples; $T_{50\%\ CO}$ for 20 % $Cu_xMg_{1-x}Cr_2O_4/Al_2O_3$ (curve *2*) is 180 °C. A sample based on MnO and LaO (Mn−La/AlSi) turned out to be less active; $T_{50\%\ CO}$ is 200 °C (curve *1*).

The relatively low catalytic activity of Mn−La/AlSi catalyst is related to the insufficiently high indicator of the pore structure of the ceramic carrier. The pore volume of this catalyst is 1.5−2 times lower compared to alumina catalyst carriers, whereas the value of A_{BET} is 5−7 times smaller.

The effect of reaction conditions for the CO oxidation reaction on the behaviour of promising catalysts was also analysed. The catalysts that showed high catalytic activity ($Pt-Pd/5\%$ $CeO_2-Al_2O_3$ and 20 % $CuCr_2O_4/Al_2O_3$) were investigated at elevated temperatures of gas flows. Figure 3 presents temperature dependences of CO conversion at a space velocity of 4000 h^{-1}. An increase GHSV between 1000 and 4000 h^{-1} leads to a temperature shift for 50 % CO conversion to-
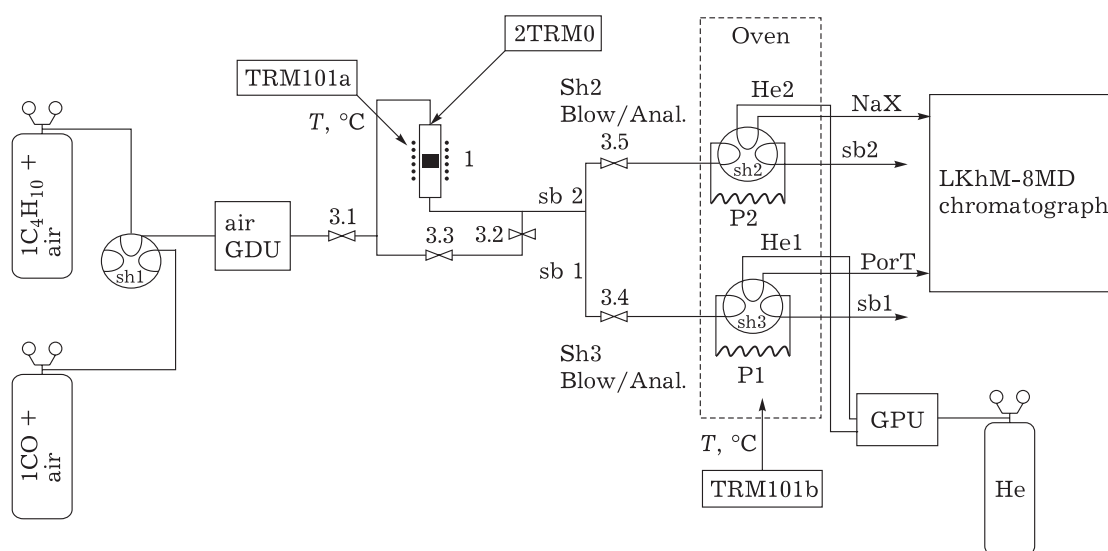

Fig. 1. Flow sheet of the setup for determining catalytic activity in hydrocarbon and CO oxidation reaction: *1* - reactor, Sh1−Sh3 − six-way valve, Z1−Z5 - stop valve, line1−line 2 − gas lines, P1−P2 − sample loop, sb1 − sb2 − to vent, NaX, Por.T − chromatography columns, GDU − gas dosing unit, GPU − gas preparation unit, TRM101 a,b − Temperature control unit, 2TRM0 − 2-channel temperature measuring instrument.
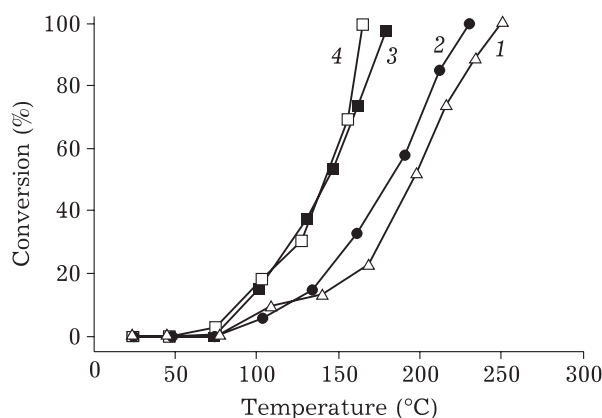
Fig. 2. Temperature dependences of CO conversion for catalysts with different chemical composition at a space velocity of 1000 h^{-1}: 1 − Mn−La/AlSi, 2 − 20 % Cu$_x$Mg$_{1-x}$Cr$_2$O$_4$/Al$_2$O$_3$, 3 − 20 % CuCr$_2$O$_4$/Al$_2$O$_3$, 4 − Pt−Pd/5 % CeO$_2$−Al$_2$O$_3$.
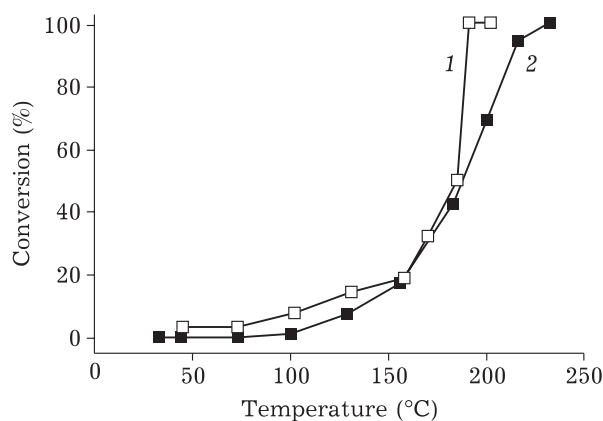

Fig. 3. Temperature dependences of CO conversion at a space velocity of 4000 h^{-1} over catalysts: 1 − Pt−Pd/5 % CeO$_2$−Al$_2$O$_3$, 2 − 20 % CuCr$_2$O$_4$/Al$_2$O$_3$.
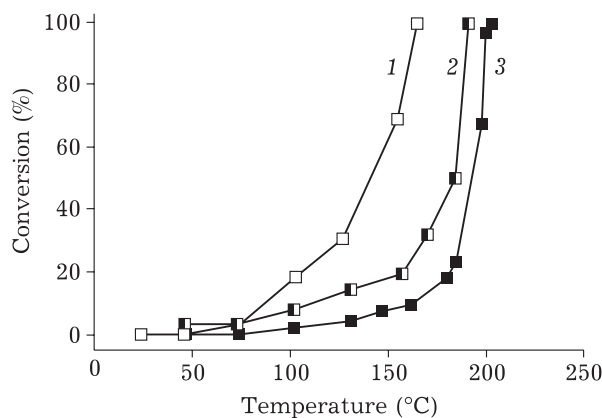
Fig.4. Temperature dependences of CO conversion at a space velocity of 1000 h^{-1} over Pt − Pd/5 % CeO$_2$ − Al$_2$O$_3$ catalyst at a space velocity, h^{-1}: 1000 (1), 4000 (2), and 10 000 (3)

wards the high-temperature region and is equal to 180 °C for these samples. There are differences in the catalytic activity of samples at temperatures of reaching 100 % CO conversion being equal to 190 and 230 °C for Pt−Pd/5 % CeO$_2$−Al$_2$O$_3$ (see Fig. 3, curve 1) and CuCr$_2$O$_4$/Al$_2$O$_3$ (curve 2), respectively.

The temperature of reaching 100 % CO conversion is of great importance to use catalysts in industrial plants for the disposal of gas emissions caused by enterprises. Catalyst operation at lowered temperatures enables to save heat resources aimed for heating the catalytic reactor.

In addition, a laboratory sample of catalysts based on noble metals was explored at a GHSV of 10 000 h^{-1}. Figure 4 presents the temperature relationships of CO conversion over Pt−Pd/5 % CeO$_2$−Al$_2$O$_3$ catalyst at various space velocities. An increase in a GHSV between 4000 and 10 000 h^{-1} leads to a slight temperature shift for

50 % CO conversion (190 °C) and 100 % CO conversion (210 °C).

According to research results, it was concluded that Pt−Pd/5 % CeO$_2$−Al$_2$O$_3$ was optimum to be used in the plant. The catalyst displays high activity preserved under service conditions in the CO oxidation reaction at various volumetric flow rates. Herewith, 100 % CO conversion is achieved at low temperatures (190−210 °C).

UNIT PROCESS SCHEME AND CATALYTIC REACTOR DESIGN

Figure 5 reports the unit process scheme to purify gaseous combustion products.

Gaseous combustion products of powder, initiating explosives and pyrotechnic compositions after the thermal treatment of substandard products in a mixture with air from the exhauste system with a temperature between −30 and 50 °C (depending on the time of a year) are fed to the catalytic plant.

Prior to feeding to the catalytic reactor, the gas is purified from particulate matter with FVP 70−40 filter and vapours of mercury, sublimates of lead and other toxic impurities that may catalyst using the carbonl filter. An FVKOM-W-CARB filter or a bulk filter with SAAFCarb MM sorbent designed to purify gases from mercury vapours may be used as a filter. Apart from Hg and Pb, carbon filters entrap nitrogen oxides and sulphur oxides, hydrogen sulphide, and organic matter including those that contain chlorine, sulphur, and phosphorus. Nevertheless, carbon filters poorly adsorb carbon monoxide (CO) and this component should be removed using an oxidation catalyst.
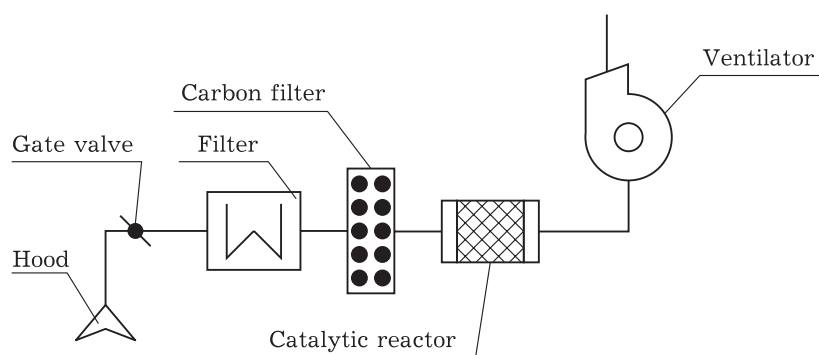
Fig. 5. Flow sheet for purifying combustion products after the thermal treatment of ammunition

TABLE 2

Major reactor technical parameters

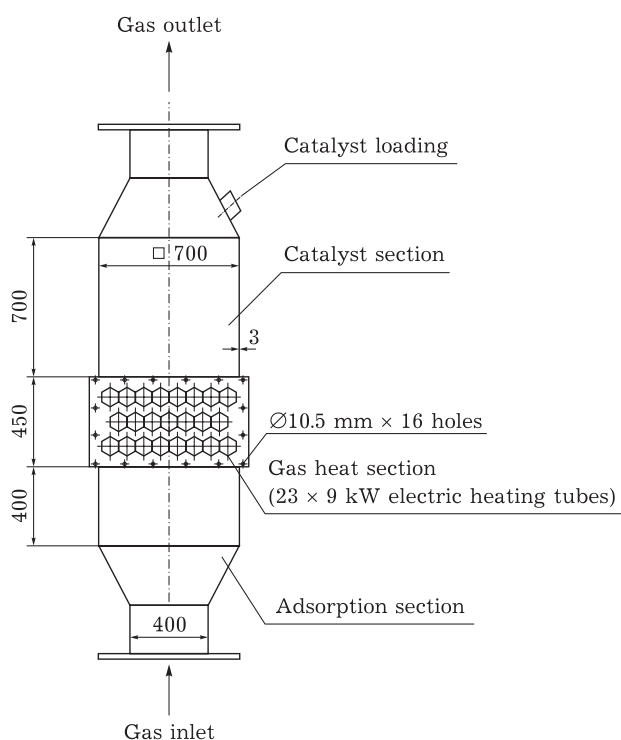| Parameter	Value
CO concentration in gas at reactor inlet, mg/m^3	200
Gas flow rate in reactor, nm^3/h	2000
Reactor operating temperature, °C	200
Reactor section, mm	700 × 700
Assembled reactor height, mm	3000
Pt–Pd catalyst charge, m^3	0.2
CO concentrationat plant outlet, mg/m^3 not more than	3

Fig. 6. Catalytic reactor.

After gas treatment from solid matter and major pollutants, the gas that contains CO is fed into the heating section of the catalytic reactor heating up till the required temperature (200 °C) with tubular heating elements. Afterwards, the gas flow is fed to the catalyst. As a result of the oxidation reaction, the concentration of CO at the catalyst outlet is reduced to the MPC level.

$$CO + 0.5O_2 \rightarrow CO_2$$
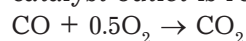
The reactor (Fig. 6) is a cylindrical or right-angle capacity with a grid, onto which the Pt–Pd catalyst for CO oxidation is loaded. The lower part of the reactor is designed to heat the gas untill the required temperature with tubular heating elements.

Table 2 reports the major technical parameters for the catalytic reactor.

CONCLUSION

Environmental issues of thermal treatment of ammunition were analysed. An opportunity to purify furnace ventilation emissions from thermal treatment using sorbent and deep oxidation catalyst was investigated. The activity of a se-

ries of catalysts was explored. Relying on the research performed, the most active catalyst, Pt−Pd/5 % CrO_2−Al_2O_3, was selected. A technology was suggested and a plant to purify ventilation emissions caused by the thermal treatment of sub-standard ammunition (cartridges, casings, and small arms primers) in an electric furnace with an inner screw was developed. The technology enables removal of vapours of mercury, lead, nitrogen oxides, and other inorganic impurities from the gases on a sorbent and CO oxidation over a solid catalyst. Currently, a plant has been assembled; catalyst loading has been performed, and tests have begun at the enterprise.

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