

TECHNICAL REPORT

on works fulfilled in the fourth quarter according to the Project # 1604

"Research of possibility of semi-conducting CdGeAs₂
making by the way of deep impurities doping"

I. Summary of Technical Progress (By task in the Work Plan)

According to the Work Plan under the Project and recommendations of the report for the III quarter, the researches in the IV quarter were directed on a solution of the following tasks:

- a) research of influence combined doping of a melt by impurities of copper (acceptor) and indium (donor) on physical properties of obtained CdGeAs₂-crystals;
- b) research of a possibility of compensating of crystals n-CdGeAs₂: In by impurity copper by the way of Cu-diffusion;
- c) clearing up of a possibility of growth of crystals CdSnAs₂ by the Chohralsii method on the "REDMET-8"-setup.

Besides during the IV quarter initiative additional researches on the following problems were performed.

- d) research of influence of doping by impurity gadolinium of crystals CdGeAs₂, grown by a HGF-method
- e) research of influence of annealing in vapour of volatile components on properties of n- and p-CdGeAs₂ with the purpose of obtaining data about a nature of intrinsic defects in this crystal.

In the framework of the above-mention tasks the following works were performed:

1.1. Synthesis of non-doped and doped by indium polycrystalline ingots of CdGeAs₂

Synthesis of CdGeAs₂-compound was carried out by one-temperature method according to two-step scheme. Technique of synthesis (and the growing) of non-doped and doped CdGeAs₂-crystals were similar to this one circumscribed in the Reports on I - II quarters of the given Project. For doping of a melt elementary In of a high purity (99.9999 %) was used. The doping of CdGeAs₂ was carried out by introduction of the impurity in charge for synthesis of the compound in the amount of 0.1 weight %. As a result mono-phase polycrystalline ingots of CdGeAs₂-compound (without microincludings of second phases) were synthesised. For the purpose of measurements of physical properties of CdGeAs₂-crystals the works on growing of monocrystals from obtained synthesised polycrystals were fulfilled. Monocrystals of CdGeAs₂ were grown by the Horizontal Gradient Freezing method. The samples cut out from large blocs of material after synthesis without recrystallisation were also used for a research of electro-physical parameters.

1.2. Measurements of electrophysical properties of monocrystals CdGeAs₂ doped by indium

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14. ABSTRACT
 This report results from a contract tasking Advanced Technologies for Optical Materials (ATOM) as follows: The contractor will develop methods of semi-isolating high-resistance CdGeAs2 crystal production by way of control doping by the impurities Cu, Au, Zn, Sc, and Se. Selection of the most promising impurities and optimization of the doping technology will allow minimization of optical losses in CdGeAs2-crystals and related to manufacture of ternary CdGeAs2 crystals with low absorption coefficient for practical purposes of nonlinear optics

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Measurements of electrophysical properties were carried out on the samples cut from large monocrystal blocks of CdGeAs₂. Size of the samples corresponded to (5-10)×(3-5)×(0.5-1.5) mm³. The contacts were made by fusing In into corresponding points of the sample at temperature 300°C.

The measurements shown that all obtained material of CdGeAs₂:In was rather homogeneous as to electrophysical parameters along the volume of the ingots. It is possible to judge about a scatter of values of some electrophysical parameters of obtained CdGeAs₂, doped by indium, on data, represented in table 1.

Table 1

Electrophysical parameters of the samples of n-CdGeAs₂ doped by indium during synthesis (concentration of In in a melt, N_n=0.1 weight %)

Sample	T, K	ρ, Om·cm	n, cm ⁻³	m cm ² /V·s
55	289.5	0.0016	1.3·10 ¹⁸	3060
	102.8	0.0016	1.3·10 ¹⁸	2980
56	297.5	0.0022	1.4·10 ¹⁸	2240
	100	0.0025	1.3·10 ¹⁸	1910
57	290	0.00127	2.8·10 ¹⁸	1770
	98	0.00128	2.7·10 ¹⁸	1780

The presence of indium in a melt during synthesis of the compound improves quality (including homogeneity) of synthesised material and allows to receive crystals of n-type of conductivity with certainty. That is the obtained result testifies to an efficiency of the techniques of obtaining of n-CdGeAs₂ by the caring of indium in the melt during CdGeAs₂-crystals synthesis.

1.3. Eelectrophysical properties of obtained CdGeAs₂ crystals doped by indium and copper

The electrophysical parameters of crystals CdGeAs₂, obtained by combined doping by In during synthesis of a material and by Cu during diffusion annealing of CdGeAs₂:In, are represented in table 2.

Table 2

Typical values of electrophysical parameters of CdGeAs₂, doped by In (In concentration in a melt is 0.1 weight %) and Cu (diffusion annealing of CdGeAs₂:In at T=500 °C, t=48 hours)

Sample	T, K	ρ, Om·cm	R, cm ³ /A·s	m cm ² /V·s	Conductivity type
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58-2	289	0.02	-30	1460	intrinsic
	80	2.6	+83	32	p

Typical values of electrophysical parameters of obtained CdGeAs₂-monocrystals, doped by indium from the melt during synthesis of the material (In concentration in a melt is 0.1 weight %) and doped by copper during recrystallization of a synthesised material by the Horizontal Gradient Freezing method (Cu concentration in a melt is 0.1 weight %) are represented in table 3.

Table 3

Typical values of electrophysical parameters of CdGeAs₂, obtained as a result of combined doping of CdGeAs₂-crystals from a melt by impurities In and Cu

Sample	Impurity concentration in a melt, weight %	T, K	ρ , Om·cm	R, cm ³ /A·s	n, cm ⁻³	m cm ² /V·s	Conductivity type
AB128#65	In:0.1, Cu:0.1	293	0.31	-	2.97·10 ¹⁷	67	p
		102	0.65	-	1.1·10 ¹⁷	88	
AB128#63	In:0.1, Cu:0.1	369	0.8	-13.4	-	17	δ
		341	0.96	+18.9	-	20	
		289	1.41	+50.6	-	36	
		90	2.95	+126.6	~5·10 ¹⁶	43	
AB128#64	In:0.1, Cu:0.1	291.5	0.05	-	1.8·10 ¹⁷	720	n+p
		90	0.07	-	1.8·10 ¹⁷	470	
AB130#66	In:0.1, Cu:0.05	290	2.3	-	5·10 ¹⁶	55	p
		100	2.7	-	2.6·10 ¹⁶	90	

The data represented in tab. 2 and 3 show that effect of compensating of donor impurity In by acceptor impurity Cu is observed, however, considerable increase of a specific resistance of the samples do not happen. As it follows from data represented in the table 1 of the report in the third quarter the specific resistance of the samples of CdGeAs₂ after synthesis and recrystallization of a synthesised material without doping or doped by zinc is much more (about 4 Omcm at room temperature and 4·10⁵ Om·cm at nitrogen temperature).

Probably in the case of CdGeAs₂:In:Cu the specific resistance is such small, because of that created by In and Cu impurity levels are shallow with a small energy of ionisation. That follows from a very small an angle of slope of the dependencies of a logarithm of specific electroconductivity (or concentration of charge carriers) from inverse temperature for CdGeAs₂-samples

doped by In and Cu. For example, from the slope of temperature dependence of specific electroconductivity for sample 58-2 values $E_1 \sim 0.012$ eV and $E_2 \sim 0.49$ eV are obtained. The value E_2 corresponds to CdGeAs₂ forbidden energy gap. The value E_1 is, probably, an energy of ionization of a level, connected with impurity copper.

1.4. Measurements of electrophysical properties of monocrystals CdGeAs₂ doped by gadolinium

The doping of CdGeAs₂ by gadolinium was fulfilled by introduction of the impurity in charge for synthesis of the compound in the amount of 0.1 weight %. For doping of a melt elementary gadolinium of a high purity (99.9999 %) was used. As a result mono-phase polycrystalline ingots of CdGeAs₂-compound were synthesised.

Measurements of electrophysical properties were carried out on the samples cut from large monocrystal blocks of CdGeAs₂. Size of the samples corresponded to (5-10)×(3-5)×(0.5-1.5) mm³. The contacts were made by fusing In.

As well as in the case of In the measurements shown that all obtained material of CdGeAs₂:Gd was rather homogeneous as to electrophysical parameters along the volume of the ingots. It is possible to judge about a scatter of values of some electrophysical parameters of obtained CdGeAs₂:Gd on data, represented in table 4.

Table 4

Electrophysical parameters of the samples of CdGeAs₂ doped by gadolinium during synthesis (concentration of Gd in a melt, $N_{Gd} = 0.1$ weight %)

Sample	T , K	ρ , Om-cm	n , cm ⁻³	m cm/V·s	Conductivity type
ANK15#59	295	0.012	$1.8 \cdot 10^{17}$	2720	n
	98	0.015	$1.8 \cdot 10^{17}$	2340	n
ANK15#62	293	0.009	$2.8 \cdot 10^{17}$	2570	n
	94	0.012	$2.8 \cdot 10^{17}$	2000	n

Similarly indium the presence of gadolinium in a melt during synthesis of the compound improves structural quality (including homogeneity) of synthesised material and moves the electrophysical parameters of the samples in the direction of n-type of conductivity. That is gadolinium is an effective donor in the given semiconductor and doping of CdGeAs₂ by impurity of gadolinium is efficient method of obtaining electron CdGeAs₂.

1.5. Research of effect of annealing in a vapour of volatile components on properties of n- and p-CdGeAs₂.

The statement of indicated experiments is connected with carried out by us the preliminary thermodynamic analysis of possible dependencies of a modification of electrophysical parameters of crystals on annealing in the vapour of volatile components (A^2 and C_4^5). The outcomes of

thermodynamic analysis are represented as the scheme in table 5, where the following indications are used. A - acceptors, D - donors, V_A , V_B , V_C - vacancy of the elements A^2 , B^4 , C^5 , respectively; A_i , B_i , C_i - interstitial atoms A^2 , B^4 , C^5 ; A_B , A_C , B_A , B_C , C_A , C_B - disorder defects; \uparrow means increase of concentration of defects or of measured magnitude, \downarrow means diminution of concentration of defects or of measured magnitude.

Table 5

The scheme of the correspondence of observable modifications of concentration and mobility of charge carriers to a modification of concentration of intrinsic structural defects at annealing in the vapour of volatile elements A^2 and C^5

Annealing in the vapour of	Observation	Possible motive	Concretization
C^5	$n - p - m -$	A^-	A_B^- , A_C^- , B_C^-
C^5	$n - p - m^-$	D^-	B_A^- , C_A^- , C_B^-
C^5	$n^- p - m -$	D^-	V_C^- , A_i^-
C^5	$n^- p - m^-$	A^-	V_A^- , C_i^-
A^2	$n - p^- m -$	A^-	V_A^- , C_i^-
A^2	$n - p^- m^-$	D^-	V_C^- , A_i^-
A^2	$n^- p - m -$	D^-	B_A^- , C_A^- , C_B^-
A^2	$n^- p - m^-$	A^-	A_B^- , A_C^- , B_C^-

The research of effect of annealing in a vapour of volatile components on properties of n- and p-CdGeAs₂ was carried out according to the method of "frozen reactions". The initial samples were located in an evacuated ampoules with pure As or Cd. Temperature of annealing was T=500 °C, time of annealing was 30 + 60 hours, the pressure corresponded to saturation pressure over pure As or Cd at 500 °C. The results of a modification of electrophysical parameters of n- and p-CdGeAs₂ are represented in tab. 6.

Table 6

The results of a modification of electrophysical parameters of n- and p-CdGeAs₂ during annealing in a vapour of volatile components (Room temperature)

Vapour	Sample	Initial properties			Annealing during 30 hours			Annealing during 90 hours		
		n , cm ⁻³	m cm ² /V·s	ρ , Om·cm	n , cm ⁻³	m cm ² /V·s	ρ , Om·cm	n , cm ⁻³	m cm ² /V·s	ρ , Om·cm
As ₄	29	+7.2·10 ¹⁵	290	3.	-7.6·10 ¹⁵	2800	0.3	+1.9·10 ¹⁶	211	1.6
As ₄	30	+6.5·10 ¹⁵	230	4.1	+2.4·10 ¹⁶	112	2.3	+1.7·10 ¹⁶	180	2.1
As ₄	54	+3.1·10 ¹⁶	90	2.3	-7.2·10 ¹⁵	2000	0.4	+3.4·10 ¹⁶	50	4
As ₄	51-1	-5.7·10 ¹⁷	2330	4.7·10 ⁻³	-5.3·10 ¹⁷	2450	4.9·10 ⁻³	-4.6·10 ¹⁷	2350	5.8·10 ⁻³
As ₄	55(In)	-1.3·10 ¹⁸	3060	1.6·10 ⁻³	-1.3·10 ¹⁸	1800	2.7·10 ⁻³	-1.2·10 ¹⁸	1600	3.3·10 ⁻³

Cd	31	$+7.4 \cdot 10^{15}$	160	5.4	$-7 \cdot 10^{15}$	580	1.5	-	-	-
Cd	53	$+3.1 \cdot 10^{16}$	100	2.1	$-3.5 \cdot 10^{16}$	240	0.8	$-2.5 \cdot 10^{16}$	660	0.4

Samples 29, 30, 54: Comparisons of initial properties with the properties obtained as a result of annealing during 90 hours in vapour As_4 shows a decrease of hole mobility and increase of hole concentration. According to the scheme, represented in the table 5, this result may be explained by increase of concentration of Cd-vacancies (acceptors) during annealing in vapour As_4 . The result, obtained at the stage of 30 hours, is unexpected and requires repetition of the experiments with more short time stages.

Samples 51-1, 55: Annealing in vapour As_4 of the samples of n-CdGeAs₂ (51-1 not doped, 55 doped by In) also shows a modification of concentration of vacancies. Downward tendency of electron concentration with increase of electron mobility is connected with a diminution of concentration of As-vacancies. Downward tendency of electron concentration with diminution of electron mobility is connected with increase of concentration of Cd-vacancies.

Samples 31, 53: Shift of electrophysical parameters in the direction of n-type of conductivity specifies on the fact, that in a case of annealing in vapour of Cd no other intrinsic defects, except vacancies, are exhibited. The increase of electrons concentration at simultaneous increase of mobility of charge carriers corresponds to a decrease of concentration of acceptors Cd-vacancies. The decrease of electrons concentration at simultaneous decrease of mobility of charge carriers corresponds to increase of concentration of the donors As-vacancies.

1.6. Research of a possibility of growth of CdSnAs₂ - crystals by the Chohralsii method

The experiments were carried out on the modified setup "REDMET-8", intended for growth of monocrystals of germanium.

At the first stage the basic task was a choice of a composition and a thickness of flux, ensuring preservation of melt composition during a crystallisation. It was found, that when use crucibles from glasscarbon (diameter of 50 mm and height of 40 mm) the satisfactory outcomes can be reached when use eutectic LiCl-BaCl₂ by a thickness of 4 - 5 mm as a flux. The experiments on growing of crystals were carried out in an atmosphere of an argon (at pressure 1.1 - 1.2 atm), the velocities of rotation of seed crystal and crucible were 5 turnovers in minute, velocity of an extraction of ingots was 3 mm / hour. It was found, that at these conditions the obtaining of monophase polycrystalline CdGeAs₂ ingots of stoichiometric composition with a diameter of 10 - 15 mm and length of 30 - 40 mm is possible. For clearing up of conditions of the obtaining of monocrystalline ingots a prolongation of researches is necessary.

II. Milestones Completed.

2.1. Experiments on doping of crystals CdGeAs₂ by impurity In during synthesis for concentration In in a melt 10⁻¹ weight % are performed. It is obtained, doping by indium improves a homogeneity of a synthesised material and ensure obtaining crystals of n-type of conductivity. Parameters obtained for CdGeAs₂: In at room temperature are the following: $\rho \sim 0.002 \text{ Ohm}\cdot\text{cm}$, $m \sim 3000\text{-}1700 \text{ cm}^2/\text{V}\cdot\text{s}$, $n \sim 1\text{-}3 \cdot 10^{18} \text{ cm}^{-3}$.

2.2. The electrophysical parameters of crystals CdGeAs_2 , obtained by combined doping by indium and by copper, were investigated. Doping by Cu fulfilled during recrystallisation of a synthesised material CdGeAs_2 : In by the Horizontal Gradient Freezing method, and during diffusion annealing with Cu of CdGeAs_2 : In. It was obtained, that the effect of compensating of donor impurity In by acceptor impurity Cu is observed, however, considerable magnification of a specific resistance of the material does not happen, probably, for the reason of small depth of location of impurity levels in forbidden band.

2.3. Experiments on doping of crystals CdGeAs_2 by impurity Gd during synthesis for concentration Gd in a melt 10^{-1} weight % are performed. It is obtained, that presence of Gd in a melt during synthesis of the compound improves a homogeneity of a synthesised material and shift electrophysical parameters of the samples in the direction of n-type of conductivity. Parameters obtained for CdGeAs_2 : Gd at room temperature: $\rho \sim 0.01 \text{ Om}\cdot\text{cm}$, $m \sim 2700\text{-}2500 \text{ cm}^2/\text{V}\cdot\text{s}$, $n \sim 2\text{-}3 \cdot 10^{17} \text{ cm}^{-3}$.

2.4. Work on a research of influence of annealing in vapour of volatile components on properties of n- and p- CdGeAs_2 is carried out. The obtained data testify about vacancy nature of intrinsic defects in this material. Besides at small times of annealing effect, demanding of a repetition of the experiments for the confirmation and explanation, was observed.

2.5. A potential possibility of growing of crystals CdSnAs_2 by the Chohralsii method is shown. One of the important technological tasks at a choice of a composition of a flux for liquid encapsulation of CdGeAs_2 melt is solved.

III. Summary of Personnel Commitments.

Common volume of expenditure of labour in the first quarter corresponds to 150 working days; including

V.G. Voevodin	- 40 w.d.;	A.I. Chernyshov	- 6 w.d.;
Yu.M. Andreev	- 15 w.d.;	O.V. Voevodina	- 16 w.d.;
S.A. Bereznaya	- 40 w.d.;	O.V. Leontieva	- 8 w.d.
Z.V. Korotchenko	- 25 w.d.;		

IV. Major Equipment Acquired

Equipment acquisition at the expense of the Project does not foresee.

V. Description of Significant Travel

Travels at the expense of the Project did not realise.

VI. Current Technical Status (on schedule, behind schedule, ahead of schedule)

Current Technical Status is on schedule.

VII. Delays, Problems, Suggestions

All work, scheduled on the given Project, are carried out in complete volume and in planned period. The series of researches were carried out additionally to the plan within the framework of the common concept of the Project.

The main results of the researches, carried out within the framework of the given project, can be summed up as follows.

7.1. Data on electrical activity of impurities Au, Cu, Zn, In, Sc, Gd when doping the crystals CdGeAs₂ from a melt are obtained.

7.1.1. Is defined, that In and Gd are the effective donors with a small energy of ionisation; Cu is an effective acceptor also with a small energy of ionisation; at last, Au, Zn, Sc are the donors with a large energy of ionisation, but ineffective. The maximum values of a specific resistance at room temperature are obtained for CdGeAs₂, doped by Au and Sc (up to 9-14 Om-cm). At the temperature of liquid nitrogen the greatest specific resistance are obtained for CdGeAs₂, doped by Zn and also for some non-doped crystals (up to 4·10⁵ Om-cm).

7.1.2. Maximum solubility of electroactive impurity is observed for Cu and In (about ~ 10⁸ cm⁻³), a little bit smaller solubility of electroactive impurity is observed for Gd (about ~ 10⁷ cm⁻³), observable solubility of Au, Zn, Sc had yet smaller magnitude.

7.1.3. The most complete researches were carried out for impurity Cu. The diffusion coefficient of Cu in CdGeAs₂ ($D=D_0 \exp(-E/kT)$ when $D_0 = 2 \cdot 10^{-3} \text{ cm}^2/\text{s}$; $E = 0.68 \text{ eV}$) and solubility of Cu in a wide interval of temperatures (for $T < 600^\circ\text{C}$ $N = N_0 \exp(-H/kT)$ when $N_0 = 6 \cdot 10^{19} \text{ cm}^{-3}$, $H = 0.3 \text{ eV}$) were appreciated. The obtained data can be used for purposeful action on parameters of crystals CdGeAs₂.

7.2. It was found, that doping by impurities Sc, Gd, In much improves structural performances of growing crystals CdGeAs₂, probably, because of clearing of a melt from uncontrollable background impurities. It is rationally to realise additional researches of this effect with the purpose of optimisation of conditions of doping.

7.3. New data on a nature of intrinsic structural defects in the crystals CdGeAs₂ are obtained. A prolongation and extension of researches on heat treatment of the crystals CdGeAs₂ in the vapour of volatile components (as model experiments for the analysis of processes defect formations in ternary chalcopyrites) is rational.

7.4. Prolongation and extension of work on growing of the crystals CdGeAs₂ by the Chohralsii method is rationally also, as this method has a series of advantages in comparison with the Horizontal Gradient Freezing method.

7.5. It is represented rationally to issue the results of carried out researches as the following team-works-publications together with the American colleagues: 1) "The diffusion coefficient and solubility of Cu in the crystals CdGeAs₂.", 2) "Doping of the crystals CdGeAs₂ from the melt.". After realisation of additional researches getting-up of a paper "The heat treatment and intrinsic structural defects in the crystals CdGeAs₂." is possible also.

Project Manager

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_____ Valeriy G. Voevodin

SPECIFICATION

CdGeAs₂-samples for delivery on the Project # 1604

Sample	Impurity	Impurity concentration in a melt, weight %	Type of conductivity	Size, mm	Note
1	undoped		n	6.35×3.0×1.33	recrystallization
2	Au	1.34·10 ⁻⁴	p	5.95×3.1×1.25	synthesis
3	Au	8.1·10 ⁻⁴	n	7.05×4.8×1.35	synthesis
4	Au	8.1·10 ⁻⁴	p	5.7×4.6×1.32	recrystallization
5	Au	1.0·10 ⁻²	n	4.9×4.75×0.99	synthesis
6	Au	1.0·10 ⁻¹	p	6.0×3.5×1.35	recrystallization
7	Sc	1.0·10 ⁻³	p	5.1×5.1×0.66	synthesis
8	Sc	1.0·10 ⁻²	n	5.4×4.9×0.78	synthesis
9	Sc	1.0·10 ⁻²	p	6.2×5.4×0.89	synthesis
10	Sc	1.0·10 ⁻¹	n	5.75×5.2×0.91	synthesis
11	Sc	1.0·10 ⁻¹	p	6.1×5.5×1.07	synthesis
12	Cu	1.0·10 ⁻¹	p	6.65×3.7×1.12	synthesis
13	Cu	diffusion, 450°C, 30 hours	p	5.5×5.45×0.67	recrystallization
14	Cu	diffusion, 550°C, 87 hours	p	5.7×5.55×0.88	recrystallization
15	Zn	1.0·10 ⁻²	p	3.75×3.7×0.83	synthesis
16	Zn	1.0·10 ⁻²	n + p	9.5×4.0×1.64	recrystallization
17	Zn	1.0·10 ⁻¹	p	7.15×2.715×0.98	synthesis
18	Zn	5.0·10 ⁻¹	n + p	8.3×4.25×1.16	recrystallization
19	Zn	1.0	n + p	6.4×3.5×1.1	recrystallization
20	In	1.0·10 ⁻¹	n	4.1×4.0×1.12	synthesis
21	Gd	1.0·10 ⁻¹	n	4.9×4.75×0.74	synthesis
22	In/Cu	0.1 / 0.1	p	5.2×2.85×1.15	recrystallization

23	In/Cu	0.1 / 0.05	p	5.3×2.75×0.98	recrystallization
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FINAL REPORT ADDENDUM

QUESTIONS & RESPONSES

(Based on communications at contract completion between
Air Force laboratory representatives and the contractor, January 2001.)

Q1. On the samples, what is meant by Synthesis vs. Recrystallization?

A1. Here these words concern to the genesis of ingots, namely: a) we used a term "Synthesis" if the processes of synthesis and following next crystallization were carried out in one technological cycle, without re-charge of the container; b) the term "Recrystallization" was used when we carried out repeated crystallization of a synthesized material (above named as "Synthesis"!) for deriving of monocrystals.

Q2: Is the impurity concentration the amount of impurity added to the initial charge or the impurity content of the final boule measured by some other unspecified method? If the latter, what is the method?

A2. The former is true. Generally, we had a possibility to carry out direct measurements of the impurity content in CGA here in Tomsk, but the expenditures on these measurements were not stipulated in the estimate of the given contract.