CRADA NO. NFE-06-00250

OAK RIDGE
NATIONAL LABORATORY
MANAGED BY UT-BATTELLE
FOR THE DEPARTMENT OF ENERGY

CRADA FINAL REPORT

Continuous CO_2 extractor and methods

CRADA NO. NFE-06-00250

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CRADA NO. NFE-06-00250

CRADA FINAL REPORT
FOR CRADA NO. ORNL-NFE-06-00250

Continuous CO2 extractor and methods

Between

UT-Battelle, LLC

And

Thar Technologies, LLC

The CRADA Final Report may describe the research done under the CRADA and/or incorporate technical data as needed to support conclusions.

Final Report Certification
for
CRADA Number NFE-06-00250

Between

UT-Battelle, LLC

and

Thar Technologies, LLC
(Participant)

Instructions:

Mark the appropriate statement in 1a or 1b below with an 'IX." Refer to the articles in the CRADA terms and conditions governing the identification and marking of Protected CRADA Information (PCI).

If no PCI is identified, the report will be distributed without restriction. If PCI is identified, the report distribution will be limited in accordance with the CRADA terms and conditions governing release of data. In all cases items 2 and 3 must be true. That is, the report cannot contain Proprietary Information and a disclosure must be filed prior to release of the report.

This certification may either be made by using this form or may be made on company letterhead if the Participant desires. A faxed copy of this completed form is acceptable.

The following certification is made for the subject final report:

1. (a) ☐ The final report contains information that qualifies as "Protected CRADA Information" (PCI). The PCI legend is printed on the report cover, and the PCI is clearly identified.

OR

(b) ☑ The final report does not contain "Protected CRADA Information." The "Approved for Public Release" legend is printed on the report cover.

2. The final report does not contain Proprietary Information.

3. By the signature below, the Participant has no objection to the public distribution of the final report due to patentable information.

For the Participant:

[signature]
(Name)

Vice President
(Title)

May 20, 2010
(Date)

ORNL-851 (9/2007)

Abstract

The purpose of this CRADA was to assist in technology transfer from Russia to the US and assist in development of the technology improvements and applications for use in the U.S. and worldwide. Over the period of this work, ORNL has facilitated design, development and demonstration of a low-pressure liquid extractor and development of initial design for high-pressure supercritical CO2 fluid extractor.

Statement of Objectives

The technical objective of this CRADA was to support transfer, to the U.S., of a unique Russian technology for continuous $C0_2$ extraction, including establishing a working relationships between US participant and the facility. This research effort was be performed under seven (7) tasks and the results are described herein:

Task 1 Collection and Confirmation of Ownership and Status of Intellectual Property (IP) and Negotiations on the IP Transfer / Assignment

Completed June, 2005

During the period of report the Contract #4595 effective June 22, 2005 between designers and MCC signed an agreement upon which MCC waived rights in the invention and transferred IP ownership to the developers (Assignment Letter # 558 dated April 16, 2004 in Log # 01-25-31). The Russian patent application was withdrawn.

A draft of a trilateral non-disclosure agreement (MCC-ORNL-Earth First Technologies) has been prepared and circulated to Legal departments of the sides. Negotiations on conveyance and distribution of property for engineering process and equipment between invention's authors and MCC top-management were carried on. The main principles of further cooperation with the US industrial partner on manufacturing process and equipment promotion to the US and Russian sales-markets were developed. The final decision on this matter will be made at the meeting with the US specialists in August 2005. The work plan was developed to confirm quality system management certification of sub-units, participating in designing and development of the continuous CO2 extraction manufacturing process and equipment in accordance with ISO 9000 requirements.

Task 2: Low Pressure Prototype Finalization and Testing

Completed October, 2005

For the reporting period, design documents have been developed to fabricate the lab-scale CO2

extraction system involving a 5-liter continuous extractor. Annex 1 to this report provides design description and design drawings.

After completion of the design activities, MCC selected a manufacturer to fabricate some parts of the extractor, loading/unloading device, and pneumatic drive. Justification for the selection was that it is a former subsidiary to a well-known spacecraft builder NPO PM which, in the production process, uses precision instruments and equipment, has a great experience in fabricating complex mechanical systems, has been certified to ISO 9000, and, for the last years, has fabricated a number of pilot CO2 extraction systems. In addition, the company's close location to the MCC simplifies the process of modifying parts and units to the extraction system and cuts down on transportation and travel expenses.

According to the design documents the Repair and Mechanical Plant (MCC) has began manufacturing the extractor and auxiliary units and parts for the lab-scale system.

Using the existing systems for sub- and super-critical CO2-extraction, MCC began to determine the extraction process parameters (caffeine extraction coefficient as a function of pressure, temperature, extraction time, pressure changes, etc.). These studies were needed for optimization of the CO2 continuous extraction process.

MCC sent to US Technical Monitor a complete package of design documentation for the unit. MCC indicated that they need to know technical requirements to the materials to be used for the demonstration to enable process development in the following task. EarthFirst Technologies initially suggested plant material and were also interested in swine waste extraction.

US Technical Monitor visited MCC in September 2005 and witnessed the existence of the documentation and parts fabricated at MCC for the prototype. Standard parts have been procured through ISTC.

The site was prepared for continuous CO2 extraction prototype assembly and demonstration. The infrastructure, including designing center, physical plant, central plant laboratory, special control equipment design office, production control department, plant engineer department were aware and prepared for the prototyping.

Paperwork needed for the transition to ISO 9000 has been prepared as well as complete prototype requirements.

Task 3: Delivery and Demonstration of Low Pressure Prototype

Completed March 2006 (with EarthFirst), demonstrated November 2006 (with Thar Technologies).

In August 2005, EarthFirst Technologies suffered damage from Katrina and in 2006, resigned as a partner from the project. A new partner has been identified – Thar Technologies Inc., and a new CRADA has been executed reorienting further work at the request of the CRADA partner.
Preparations for the prototype delivery started ahead of time, in June 2005. Russian export control authorities were notified, and the plan has been developed for the exports of 2 items:

- Extractor;

- Design documentation

For the customs extraction plant export registration the following information has been collected:
1. Russian TN VED code is being selected. If the "ready-mounted plant" cannot be classified under one code in accordance with TN VED it will be necessary to declare each part of the abovementioned plant separately. If the selected TN VED code falls into double assigned products control list, it will be required to receive license or competent authorities report to the effect that this commodity has no military function.

2. The full name of product is being chosen with all specifications (technical, qualitative, quantitative, GOST, brand etc.).
3. Terms of delivery are being discussed in accordance with Incoterms, transport, packing, payments, frontier customs point name, which will be used for plant export.

For design documentation export the following is being selected:

- Russian TN VED code;
- The full product name with the edition type indication – in sheets, booklet etc. The confirming documents showing intellectual activities results will be:

acceptance/commission reports, license contract/assignation contract, invoices. The delivery has not been implemented because the industrial partner – EarthFirst Technologies resigned from the project.

Thar Technologies Inc. requested removal of the ISO requirements from the project and reconfigured the project with the emphasis on design. With a significant delay, ISTC delivered parts procured for the extractor prototype, and the prototype has been assembled, preapproved by Rosatom, and prepared for the demonstration at MCC.

In September 2006, MCC developers attended an international meeting on CO2 extraction in Japan and presented their papers on the process optimization.

US delegation (Y-12 GIPP Program Manager, US Technical Monitor and Thar Technologies representative) attended the demonstration of the low-pressure prototype performance at MCC. Whereas mechanics of the system deserved high marks and is unique, fluidics had a number of problems which Thar specialists asked to address in order to make a decision on the imports of the prototype into the US. In overall, Thar was very positive about the project and decided to continue co-development of a high-pressure extractor.

Task 4: Design of High Pressure Prototype The three parties (ORNL, MCC and Thar) have been activel y corresponding and exchanging opinions on the design of high-pressure system. In November 2006, MCC specialists provided Thar, through US Technical Monitor, with their drafts for the high-pressure extractor. Thar completed the review of the design proposed by MCC and offered it own blueprint and concept of the high-pressure valve. US Technical Monotor cleared the document through Export Controls and sent it to MCC with the detailed tasks on specific technical items. MCC reported several major adjustments essential for the expansion of the codevelopmental work with Thar , most notable establishing of a protocol of a limited access to Thar IP (need-to-know procedure). The Russian side has sent some drafts prepared based on Thar requests to ORNL in 2009 attached to this report). By that time, ORNL was no longer involved with the project

Task 5: High Pressure Prototype Finalization and Testing

The US delegation consisting of two representatives of Thar Technologies and ORNL Technical Monitor traveled to Russia to observe demonstration of the high-pressure extractor (figure on previous page). The demonstration failed as the extractor kept leaking and did not produce any measurable results

Task 6: Negotiation and Execution of Benefit-Sharing Agreement

In April 2006, Thar and MCC signed mutual NDA (in addition to ORNL-Thar and ORNL-MCC PIAs), and US Technical Monitor verified the contents and sent the final version to MCC. In November 2007, Thar has visited MCC but the Russian side did not engage in those discussions.

Task 7: Delivery and Demonstration of High Pressure Prototype

As ORNL function on the project expired, this task was handled by International Science and Technology Center (ISTC) in Moscow, a DOE-appointed POC for the project. ISTC has been involved in resolving bureaucratic requirements for payment of grants and financial reports for the final two quarters of the project 2008 (Q14, Q15) and then for extension of the project through September 30, 2009. The proposed schedule was compact with many needed actions to take place culminating in a demonstration of the low-pressure extractor in Pittsburg in late November or in the first quarter of 2010. Later, in the summer of 2009, the protocol action plan was revised and deadlines were push ed back about 1-2 weeks for most project financial actions. No final actions on the project followed.

Benefits to the Funding DOE Office's Mission

The project was funded by the Department of Energy (DOE) National Nuclear Security underemployed Russian former weapons scientists in the development of civilian technologies thus meeting goals of non-proliferation.

Technical Discussion of Work Performed by All Parties

Technical work performed in Russia under ORNL management included development of a low-pressure liquid CO2 extractor, process and system that have been demonstrated to the US delegation. Technical work in Russia also included 6 d rafts of design for a high-pressure extractor.

Technical work by Thar included design of high-pressure fluid extractor which the company intended to fabricate and test at the Russian facility.

Subject Inventions (as defined in the CRADA)

None.

Commercialization Possibilities

Commercialization of Russian technologies in the US remains a challenge due to a number of obstacles, including major differences in business rules and models and lack of models and support mechanisms on both sides.

Plans for Future Collaboration

ORNL kept collaborating with Thar Technologies on a number of projects, submitted several joint research proposals, entered into an M&A and other agreements. This project led to an establishment of solid, trusting relationships based on mutual research interests between Thar Technologies/Thar Process and ORNL.

Conclusions

Commercialization of Russian technologies in the US remains a challenge due to a number of obstacles, including major differences in business rules and models and lack o f models and support mechanisms on both sides.

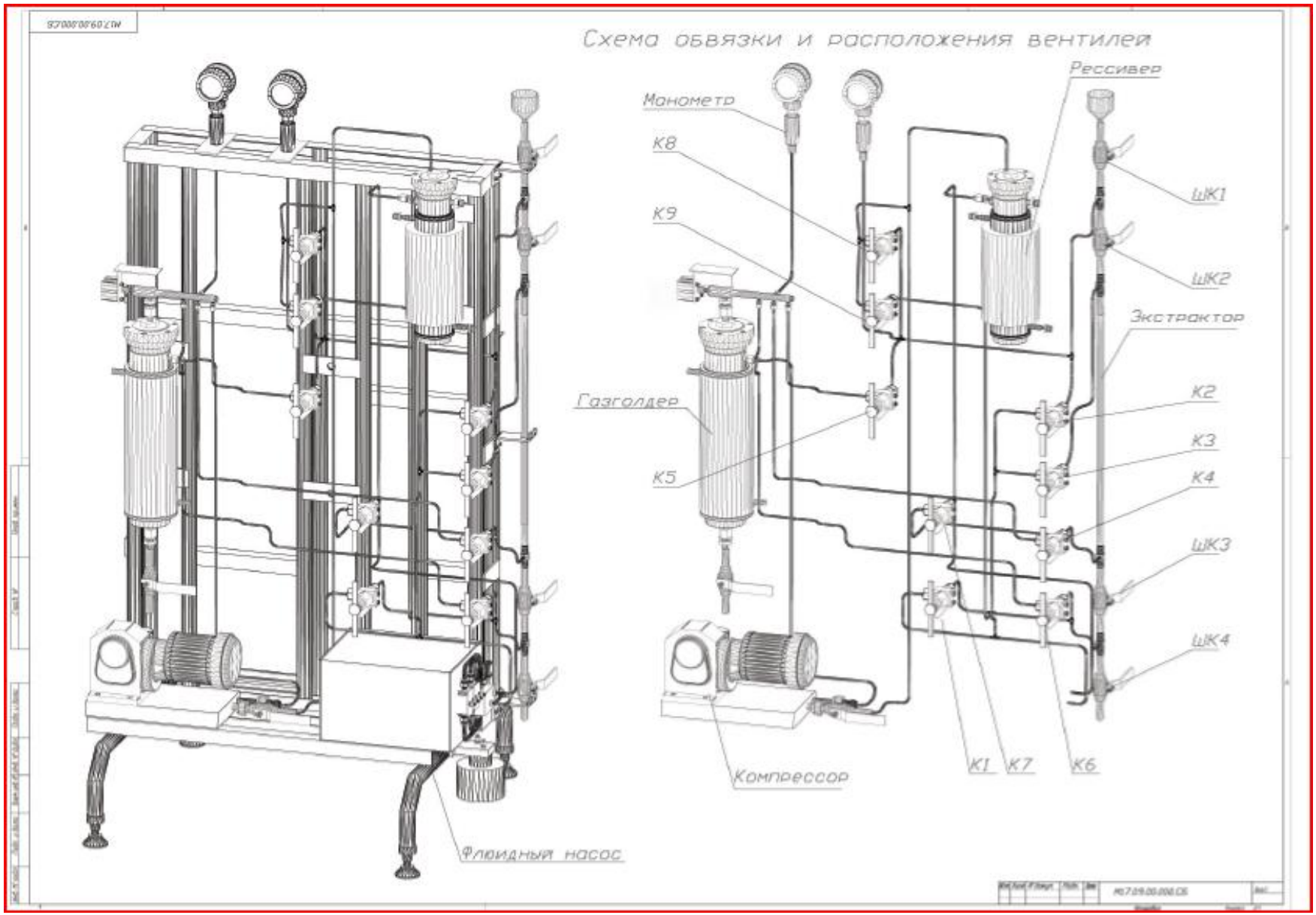
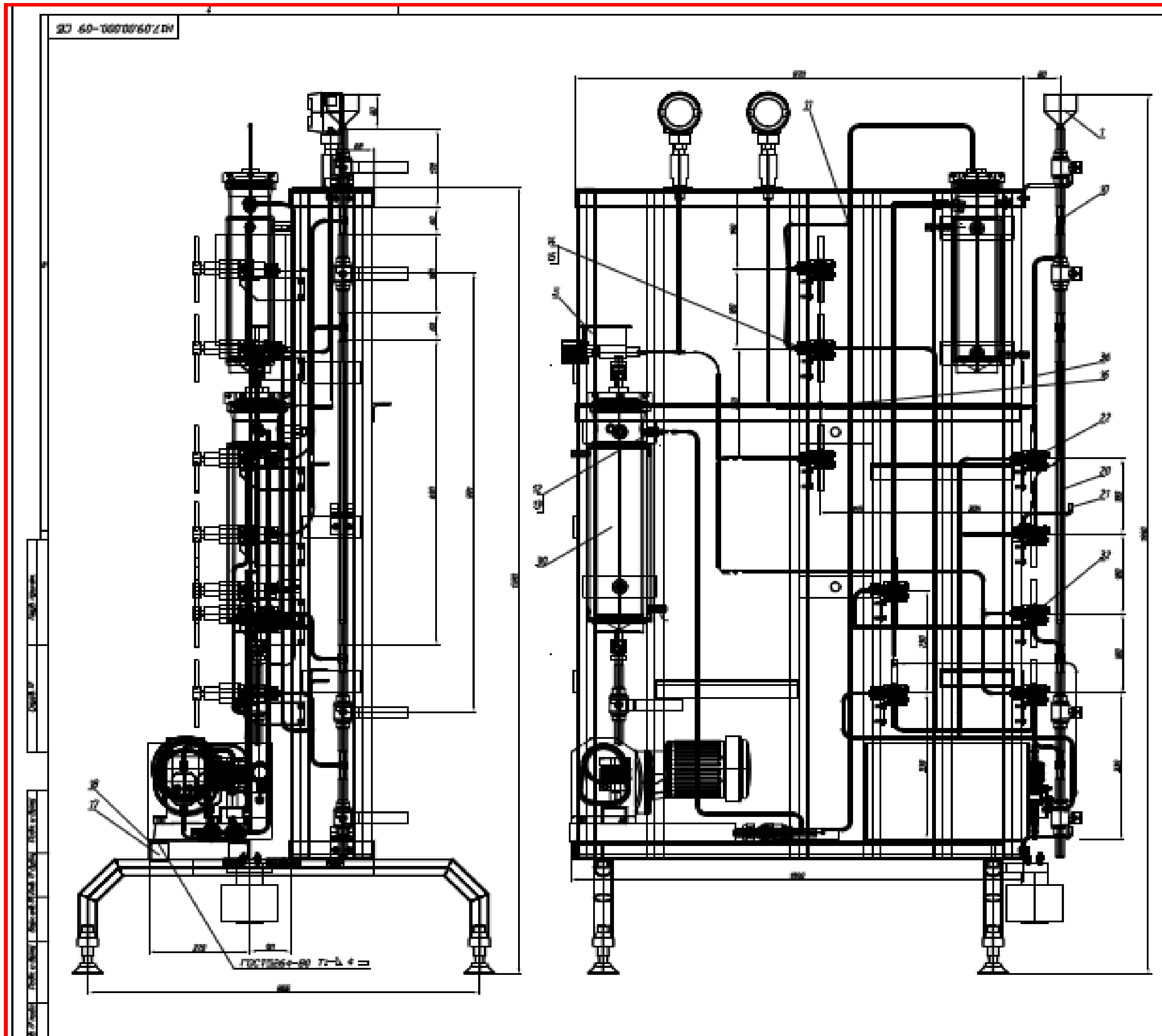


Схема обвязки и расположения вентилей
Рессивер
Манометр
К8
К9
ШК1
ШК2
Экстрактор
Газгольдер
К5
К2
К3
К4
ШК3
ШК4
Компрессор
К1
К7
К6
Флюидный насос



ТЕХНИЧЕСКАЯ ХАРАКТЕРИСТИКА

1.Рабочая среда — Диоксид углерода
температура среды, град С — -30
рабочее давление в камере, МПа(кгс/см2) — 20(200)
2. Объем рабочий, л — 0,18
3.Шлаз — Кран шаровый
4.Основной конструкционный материал — Сталь 12Х18Н10Т

ТЕХНИЧЕСКИЕ ТРЕБОВАНИЯ

1. На экстрактор распространяются Правила устройства и безопасной эксплуатации технологических трубопроводов ПБ03-585-03, Группа – Б, Категория – II
2. Качество и свойства основных и сварочных материалов подтвердить сертификатами
3. Монтаж экстрактора на раму производить по сборочному чертежу установки.
4. Экстрактор испытать на плотность пробным гидравлическим давлением 25 МПа (250кгс/см2). Испытание на герметичность провести в составе установки давлением, равным рабочему.
5. Краны шаровые испытать на работоспособность. Переключение должно выполняться без рывков и заеданий.
6. Приемочные испытания производить в составе установки в соответствии с программой и методикой испытаний установки.
7. Размеры для справок
8. Остальные технические требования по ОСТ 95.277-92.
9. Длину трубок поз. 36 определить после выполнения проекта обвязки экстрактора
9.1 Сварка ручная электродуговая в среде аргона по ГОСТ14771-76 Присадочная проволока Св-05Х19Н9 ГОСТ2246-70
10 Доработку рамы выполнить по месту. Дополнительные элементы крепить сваркой или на болтах.
10.1 Сварка ручная электродуговая по ГОСТ5264-80
Электрод Э42 ГОСТ9467-75
Электрод ОЗЛ-36 ГОСТ9466-75

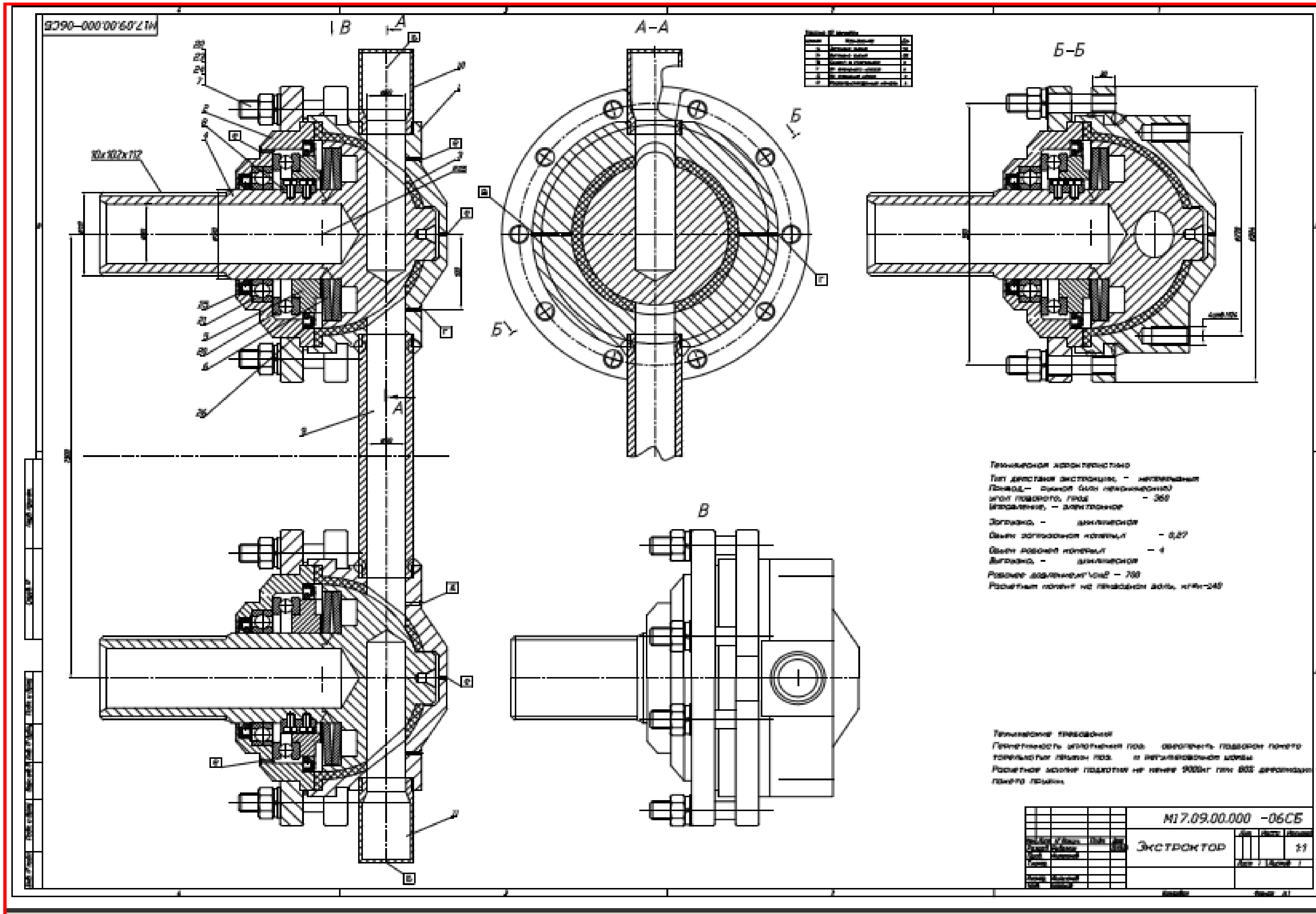
Н17.09.00.000.-09 СБ

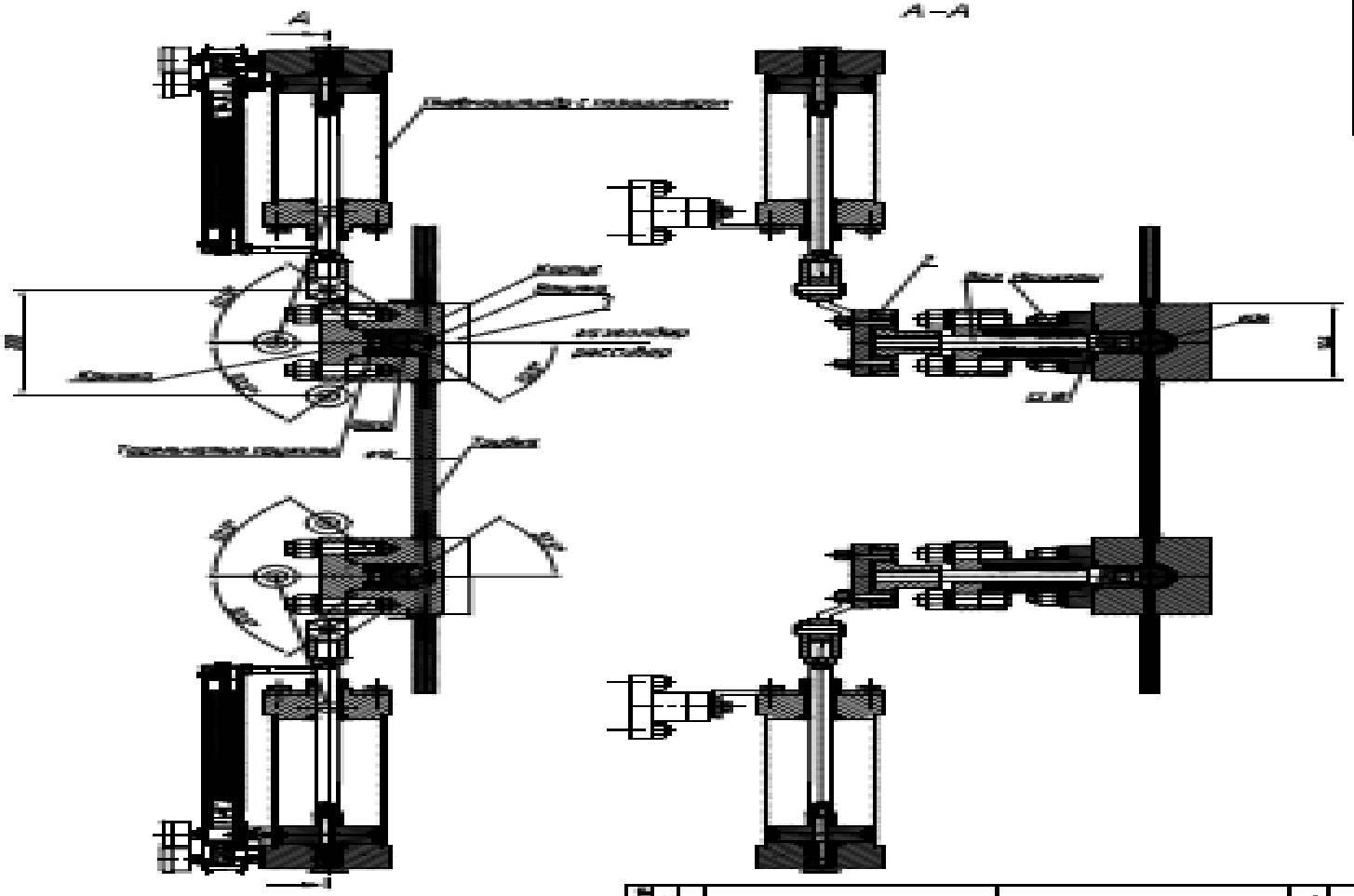
Установка экстракционная

Лит.	Масса	Масштаб
		1:1

Лист 1 Листов 1

Копировал Формат А1


М17.09.00.000 -06СБ
В
А
А-А
Б-Б
Б
10х102х112
Техническая характеристика
Тип действия экстракции, – непрерывный
Привод,– ручной (или механический)
угол поворота, град – 360
Управление, – электронное
Загрузка, – цилиндрическая
Объем загрузочной камеры,л – 0,27
Объем рабочей камеры,л – 4
Выгрузка, – цилиндрическая
Рабочее давление,кг\см2 – 700
Расчетный момент на приводном валу, кгМм-240
Технические требования
Герметичность уплотнения поз. обеспечить подбором пакета тарельчатых пружин поз. и регулировочной шайбы
Расчетное усилие поджатия не менее 9000кг при 80% деформации пакета пружин.
Экстрактор
1:1



Обозначение	Наименование	Кол.	Примеч.
	Сборочные единицы		
	Электрооборудование	1	
	Лист	1	

М17.09.00.000–01 ВО

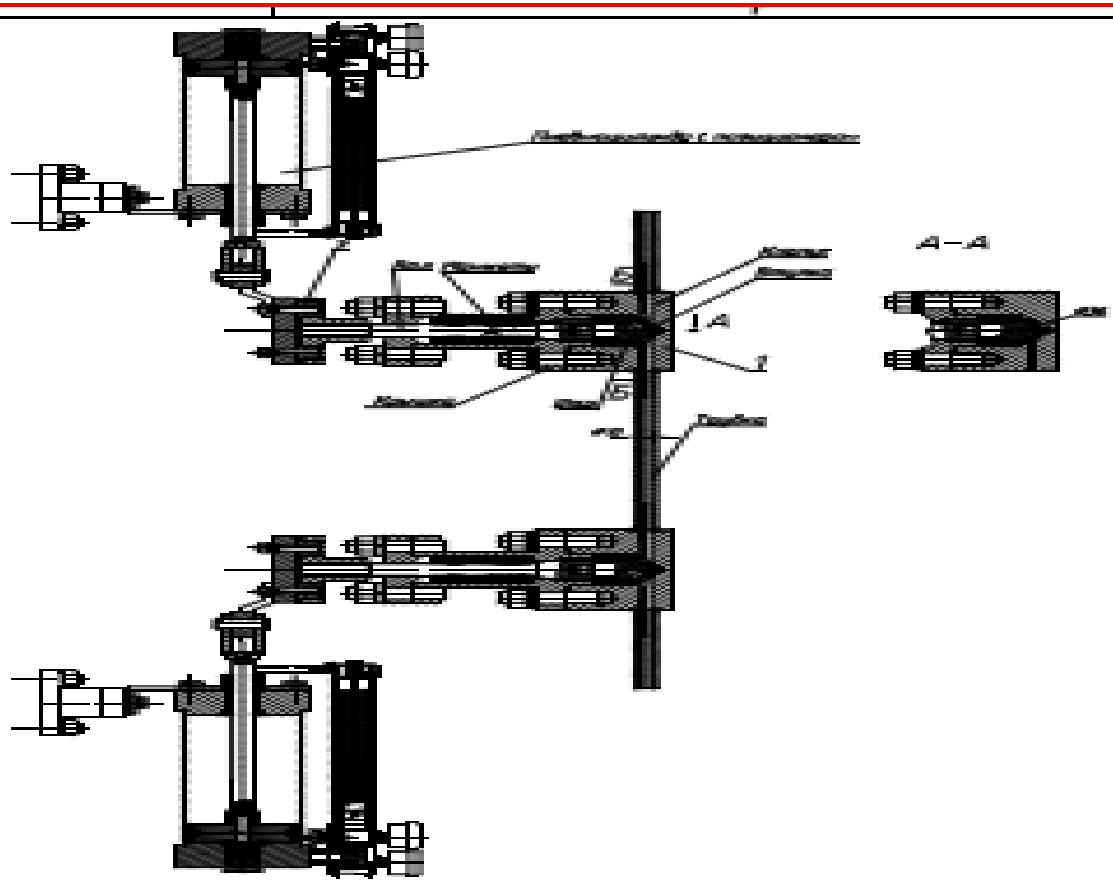
Экстрактор

Лит. | Масса | Масштаб

1:1

Лист 1 | Листов 1

Копировал | Формат А2



Обозначение	Наименование	Кол.	Примечание
	Сборочные единицы		
	Электрическая колодка	2	
	Муфта	2	

М17.09.00.000–02 ВО

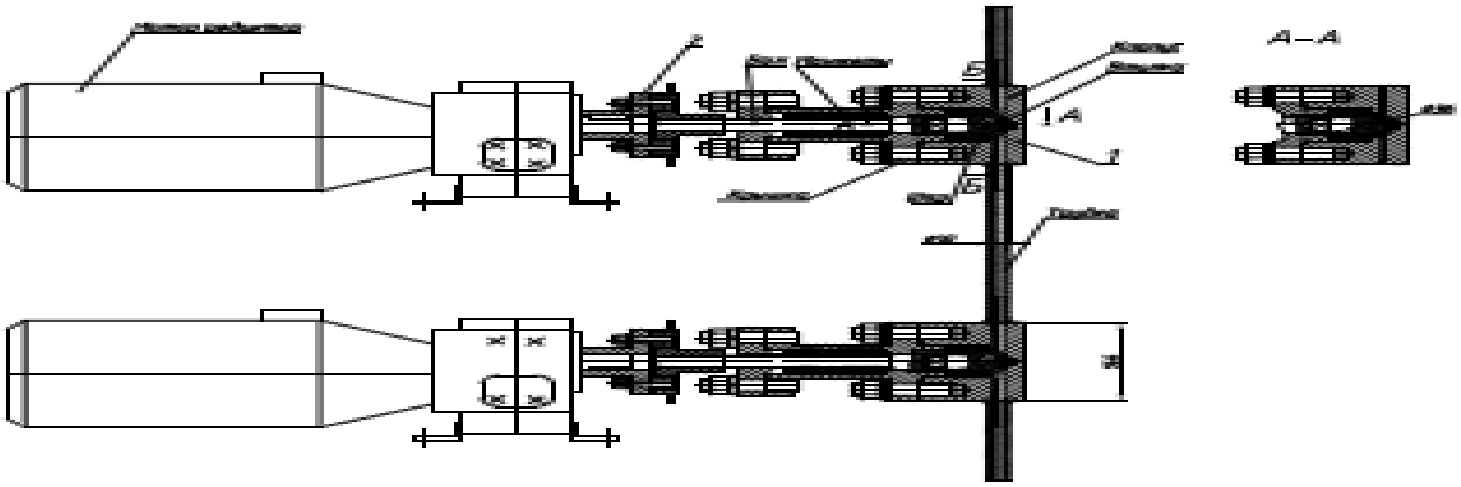
Экстрактор

Лит. | Масса | Масштаб

1:1

Лист 1 | Листов 1

Копировал | Формат А2



Обозначение	Наименование	Кол.	Примеч.
	Сборочные единицы		
	Электрическое исполнение	2*	
	Кабель	2*	

М17.09.00.000–03 ВО

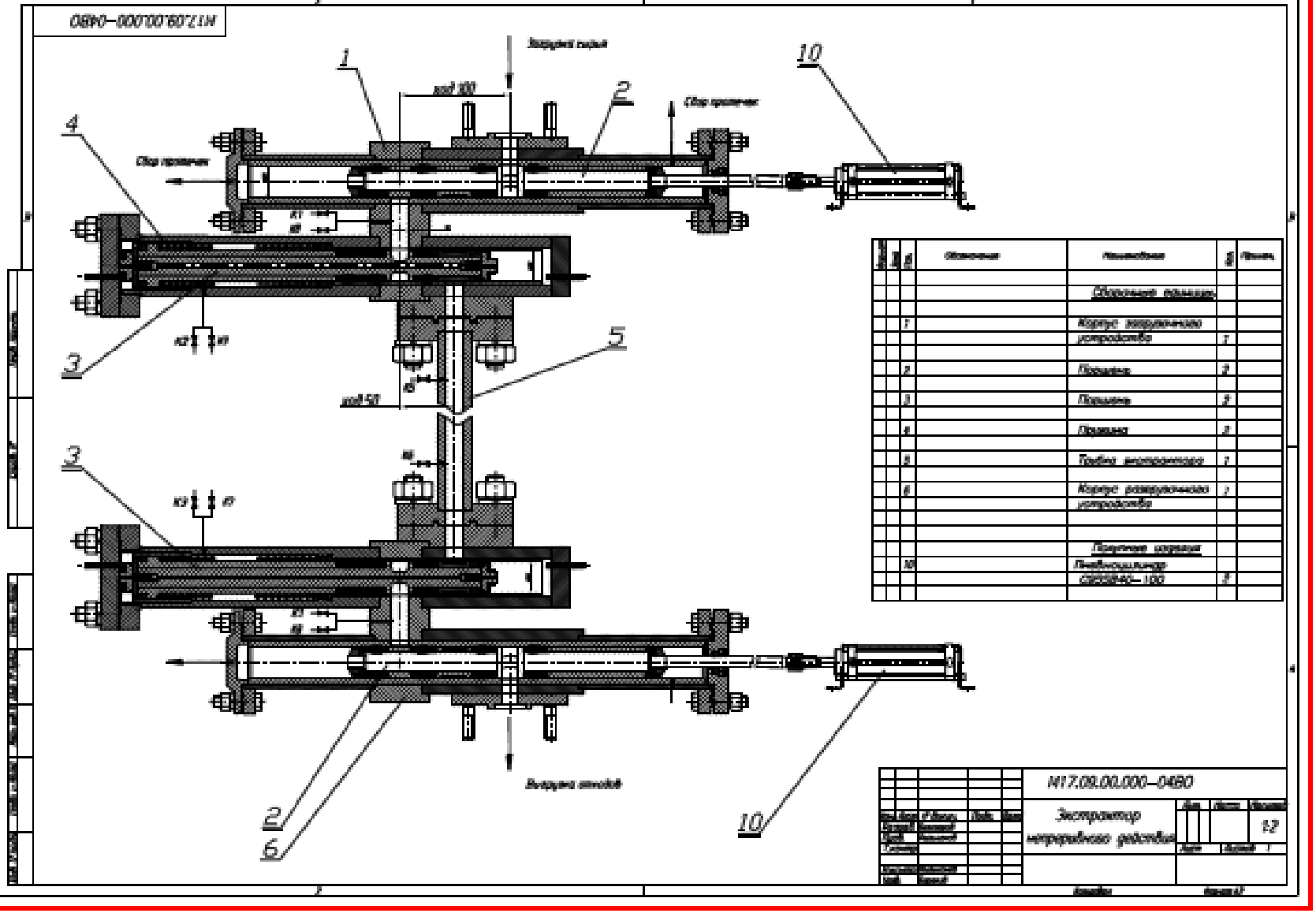
Экстрактор

Лит. | Масса | Масштаб

1:1

Лист 1 | Листов 1

Формат А2



Поз.	Обозначение	Наименование	Кол.	Примечание
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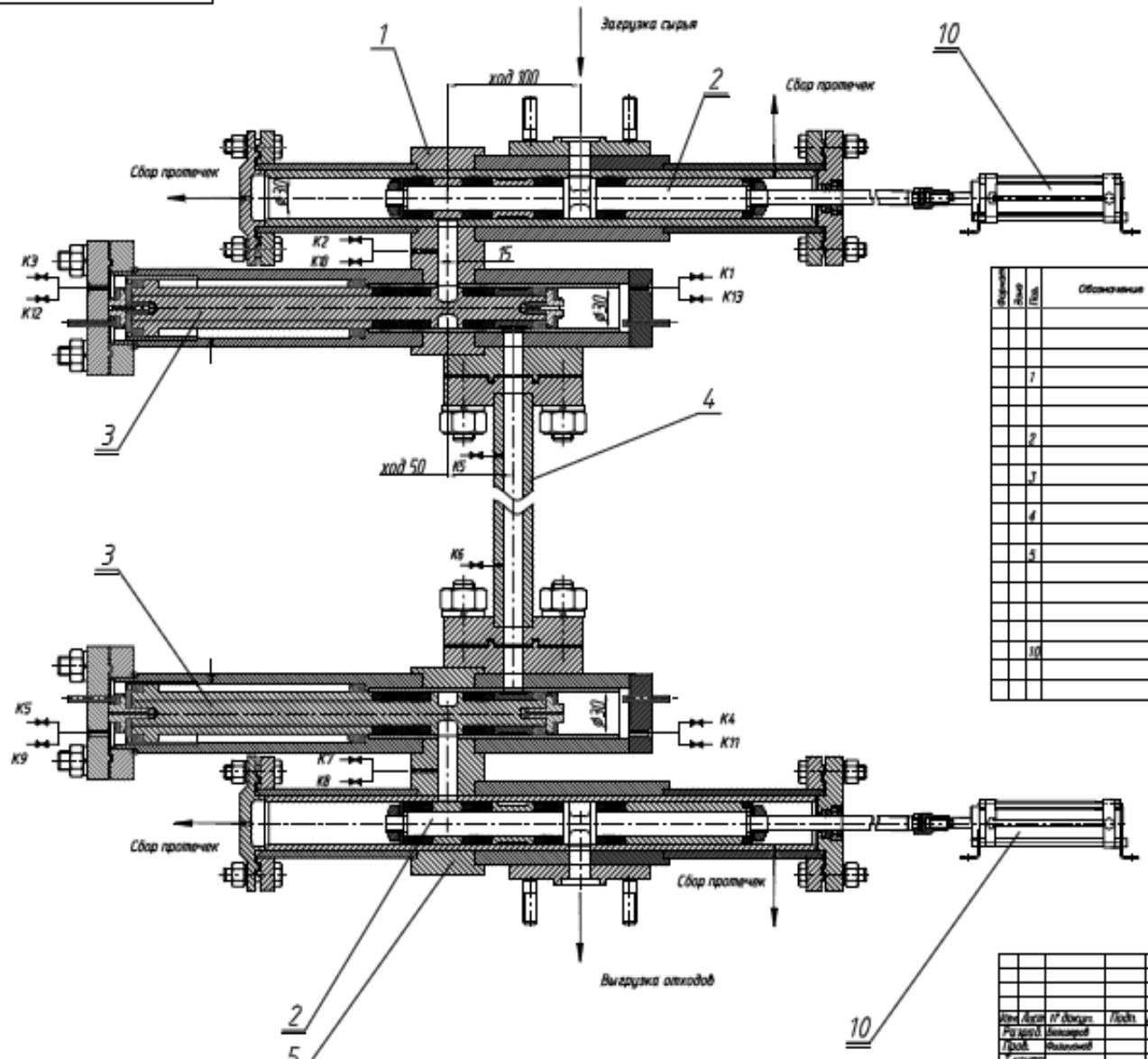
2		Поршень	2	
3		Поршень	2	
4		Пружина	2	
5		Трубка экструзионная	1	
6		Корпус разгрузочного устройства	1	
		Покупные изделия		
10		Пневмоцилиндр СДОЗВ40–100	2	

				М17.09.00.000–04ВО		
Разраб.				Экструдер непрерывного действия	Лит.	Масса	Масштаб
Пров.							1:2
Т.контр.					Лист	Листов 1
Н.контр.						
Утв.						

Копировал Формат А3



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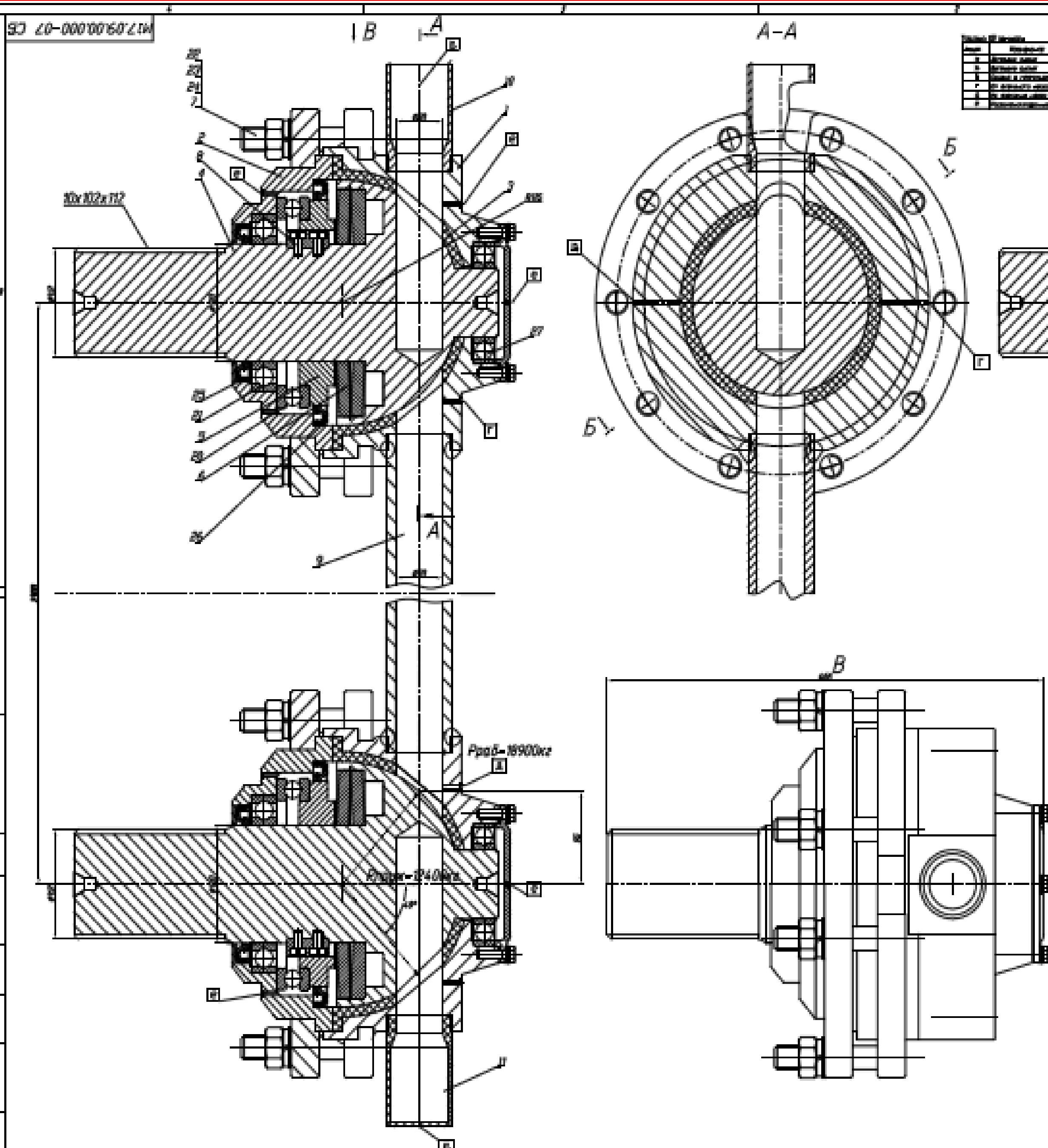
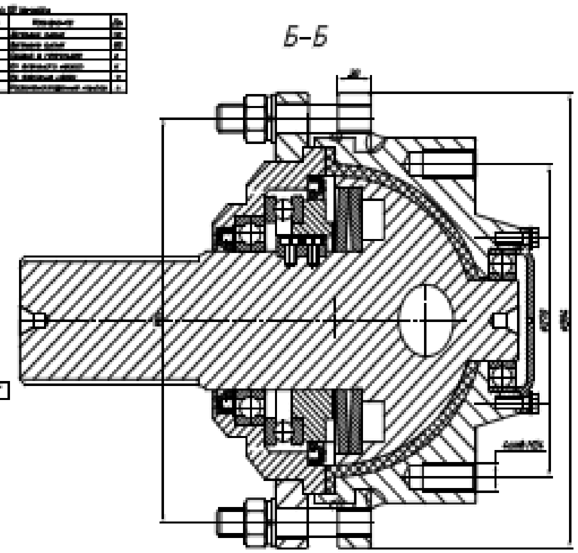
		2		Поршень	2	
		3		Поршень	2	
		4		Трубка экстрактора	1	
		5		Корпус разгрузочного устройства	1	
				Покупные изделия		
		10		Пневмоцилиндр С95SB40–100	2	

Изм.	Лист	№ докум.	Подп.	Дата	М17.09.00.000–05ВО	
Разраб.	Бекмаров				Экстрактор непрерывного действия	Лит. / Масса / Масштаб 1:2
Пров.	Филимонов					Лист / Листов 1
Т.контр.						
Н.контр.	Филимонов					
Утв.	Баранов					

Копировал Формат А2





Краткий расчет

Площадь 26,84см2

Давление 26,84х700=18900кг

Давление пяты 18900хcos49=12400кг

Усилие трения 18900х0,04...0,3=756...5670кг
где коэффициент трения фторопластов по стали= 0,04...0,3

Расчетный оводочный момент трения (756...5670)х0,095=(72...539)кг х метр

Пневмоцилиндр диаметр 250мм.

теоретическое усилие при 0,5Мпа.- 1460кг

Развиваемый момент на рычаге L=400мм. составит 598кг х м

Техническая характеристика

Тип действия экстрактора, - непрерывный
Привод,- пуансон (или механический)
угол поворота, град - 360
Управление, - электронное

Загрузка, - цилиндрическая

Объем загрузочной камеры,л - 0,27

Объем рабочей камеры,л - 4
Выгрузка, - цилиндрическая

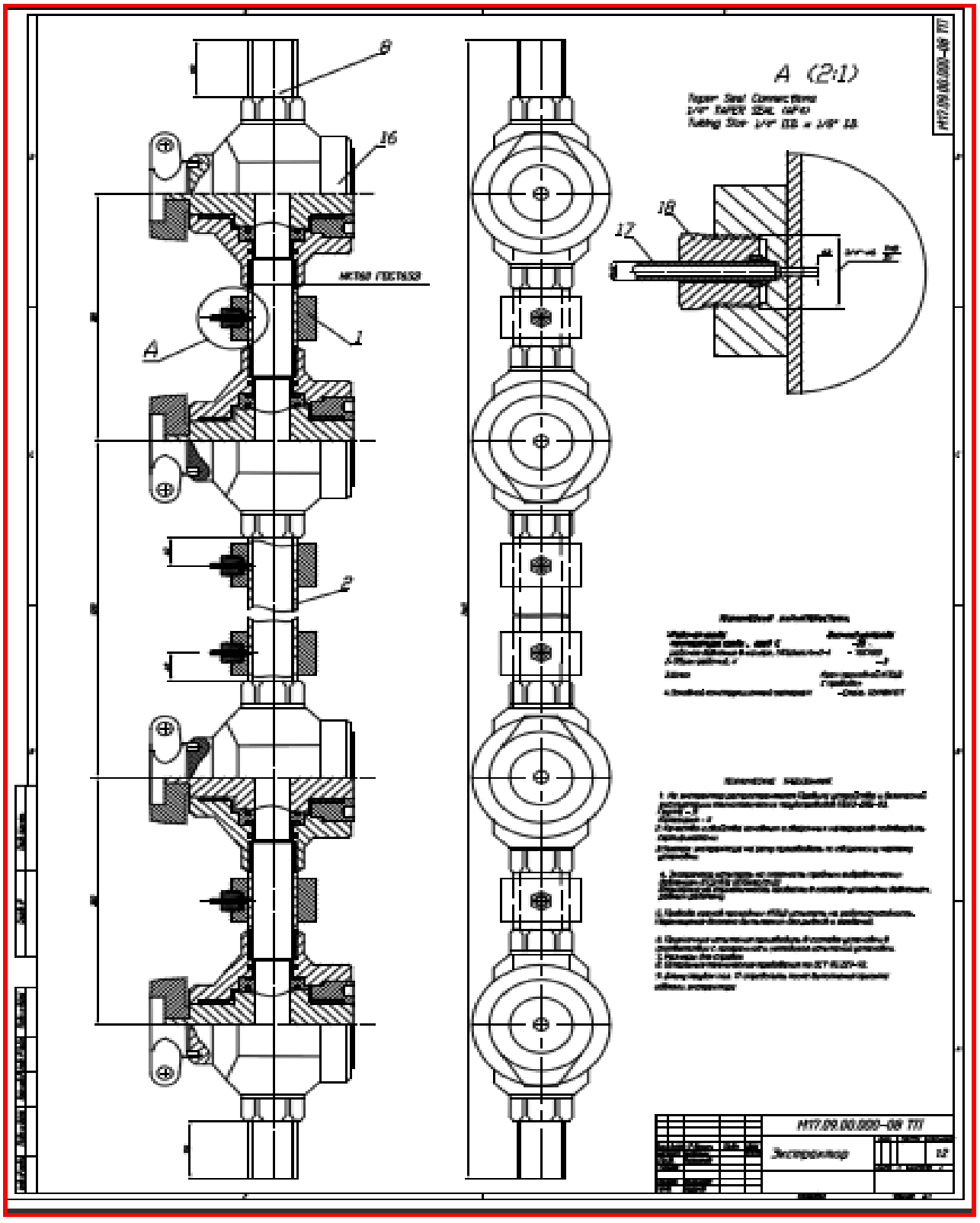
Рабочее давление,кг\см2 - 700

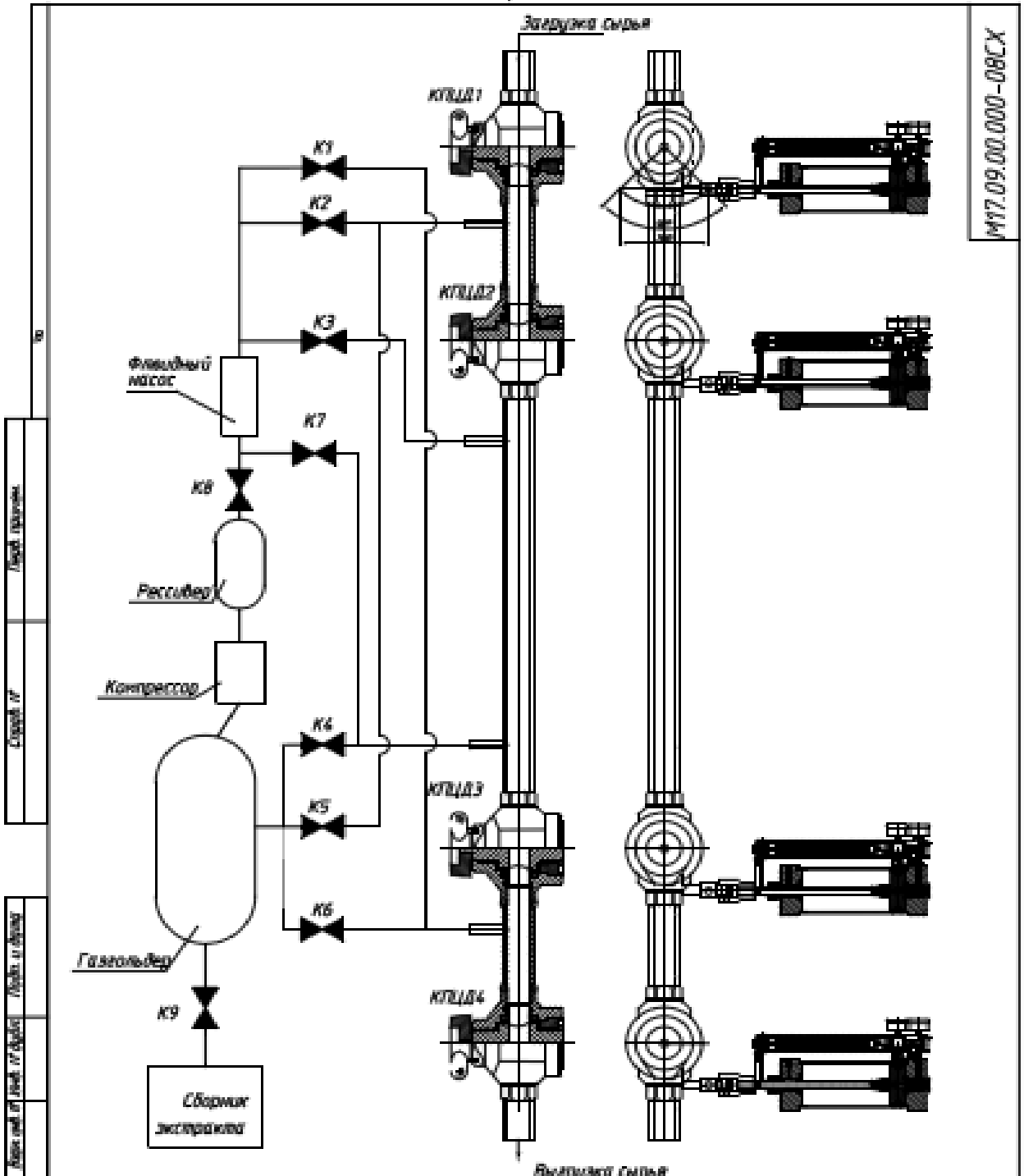
Технические требования

Герметичность уплотнений поз. обеспечить подбором пакета тарельчатых пружин поз. и регулировочной шайбы

Расчетное усилие подсжатия не менее 12500кг при 80% деформации пакета пружин.

М17.09.00.000-07 СБ	
Экстрактор	1:2


8
16
1
2
A
A (2:1)
17
18



М17.09.00.000-08СХ

						М17.09.00.000-08СХ		
Изм.	Лист	№ докум.	Подп.	Дата				
Разраб.		Рыбаков			Экстрактор Схема обвязки	Лит.	Масса	Масштаб
Пров.		Рыбаков						1:1
Т.контр.						Лист 1	Листов 1	
Н.контр.		Рыбаков						
Утв.		Бурков						

Копировал Формат А3

Инв. № подл.	Подп. и дата	Взам. инв. №	Инв. № дубл.	Подп. и дата

Справ. №	Перв. примен.

РШЕ М17.09.00.000–06

Копировал

Формат А4