

Vacuum technology

Vacuum compatible materials

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What purpose?

- Construction materials
- Sealings
- Pump consumables, oils, greases
- Etc.

What materials?

- Metals: stainless steel, copper, brass, aluminum, bronze, gold, tungsten
- Plastics-elastomers: Teflon, Viton, buthyl-rubber, silicone
- Glass: borosilicate (+ quartz, sapphire and ceramics)
- Oils: aliphatic, ester, silicone, ether, fluorinated

What requirements?

- Vapor pressure
- Vapor pressure of decomposition products
- Outgassing
- Diffusion
- Temperature limits (liquid He – few 1000 °C)
- Electric and heat insulation
- Electric and heat conductivity
- Phys-chem compatibility
- Practical parameters (maintainability, availability, price)

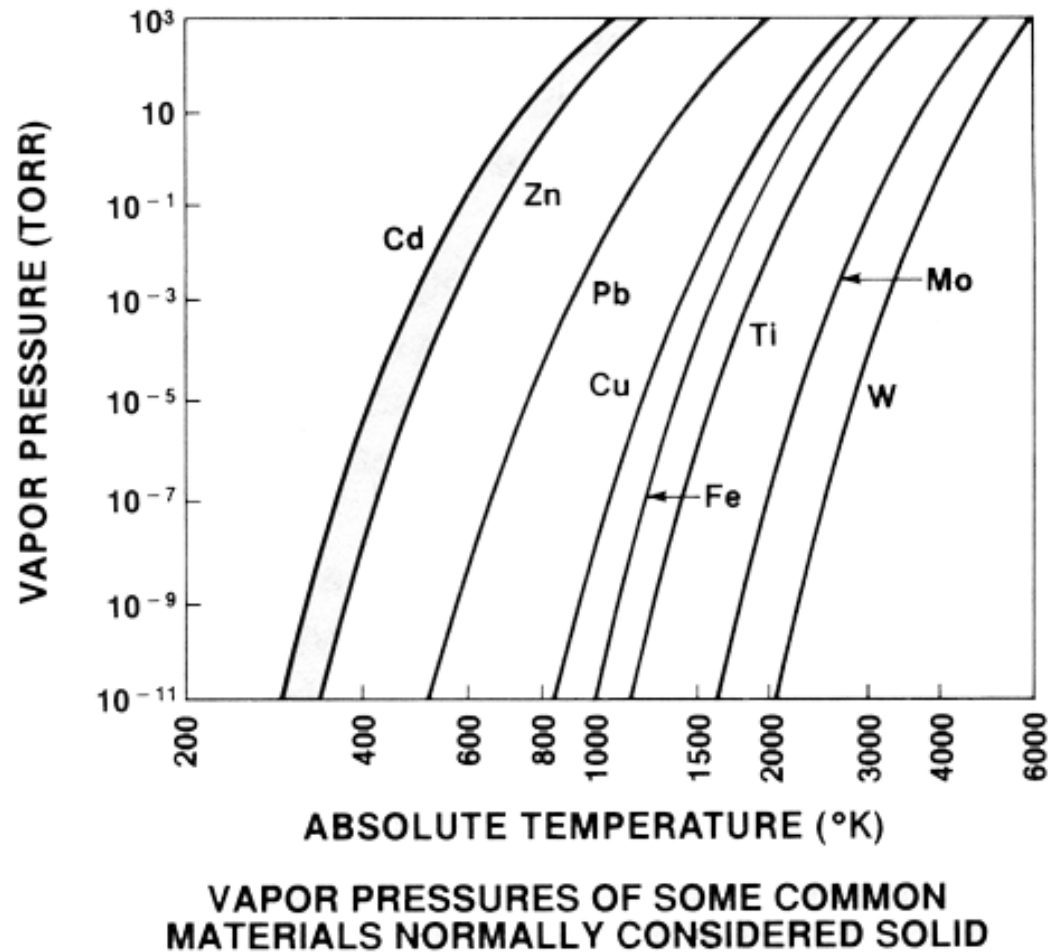
Metals

Everything has vapor pressure

- Mercury
- Zinc (brass)
- Cadmium
- Lead (soldering)
- Titanium
- Tungsten



New and used tungsten filament incandescent lamp



Metals

Stainless steel

- AISI/SAE 304 Most prevalent (Fe, <0.8% C, 17.5-20% Cr, 8-11% Ni, <2% Mn, <1% Si, <0.045% P, <0.03% S), Inox, 18/8, MSZ KO 33, DIN W.Nr. 1.4301, ISO 3506 A2, spoon :-)
austenitic: no cold brittleness, no ductile-brittle transition (martensitic, ferritic has), -269 °C - 300 °C OK.
- With some Mo: AISI/SAE 316, DIN W.Nr. 1.4401, ISO 3506 A4, „Marine grade”
- With some Ti: MSZ KO 36 Ti, AISI/SAE 321, DIN W.Nr. 1.4541, for higher temperatures. And a lot of other type.

Metals

Stainless steel

- Advantages: no rust :-) Rust can adsorb a lot of water. Good chemical compatibility. Can be welded. Pretty good mechanical properties. In general not magnetic (to be sure: μ -metal).
- Disadvantages: Price (not that high today). Soft. Not good to machine. Beware of fingerprint: eats it! Halogenides, acids, fingerprints corrode it. Nut and bolt can weld to each other.
- Kovar (54 % Fe, 29 % Ni, 17 % Co), Fernico feed through (can recrystallize in cold).
- Carbon steel not: rust. Even stainless steel is to be polished (weldings!)
- Casting not OK: porous, should be mechanically worked (forging)

Metals

Steel outgassing after 1 hour

- Rusty steel: 4.08 microliter/(sec*foot²)
- Clean mild steel: 0.50 microliter/(sec*foot²)
- Clean stainless steel: 0.16 microliter/(sec*foot²)

Metals

Steel outgassing

Approximate outgassing rate K_1 for several vacuum materials, after one hour in vacuum at room temperature.

Material	K_1 (mbar l s ⁻¹ cm ⁻²)
Aluminium (fresh)	9×10^{-9}
Aluminium (20 h at 100 °C)	5×10^{-14}
Stainless steel (304)	2×10^{-8}
Stainless steel (304, electropolished)	6×10^{-9}
Stainless steel (304, mechanically polished)	2×10^{-9}
Stainless steel (304, electropolished, 30 h at 250 °C)	4×10^{-12}
Perbunan	5×10^{-6}
Pyrex	1×10^{-8}
Teflon	8×10^{-8}
Viton A (fresh)	2×10^{-6}

Metals

Copper, bronze, brass

- Red copper: soft, good electric and heat conductivity. Oxygen-free quality (OFHC - Oxygen Free High Conductivity). Hard to machine. Sealing rings. Mechanical hardening.
- Bronze: Good machinability, good for small parts even in UHV (bolt-nuts).
- Brass: Dangerous! Zinc has large vapor pressure, RT 10^{-8} not compatible with UHV. Good for casting and machining, lot of parts are made of it (nickel-coating)
- Beware: there is such bloody thing as “zinc bronze”. Composition of copper alloys is always to be checked.

Metals

Aluminum, gold, tungsten

- Aluminum: high purity for sealing rings (soft). 7075 (ZnMg(Cu)), 6061 (SiMg), 4032 (Si) other alloys for structural material. Welding, machining. Temperature below 450 C. No brittle transition on cold. Casted parts are porous. Halogenides, acids/bases, Hg, fingerprints corrode. Good heat and electric conductivity.
- Gold: 24 karate pure for sealing rings.
- Tungsten: heating filament, electric feed through compatible with borosilicate. Oxygen eats away hot. Rhenium, canthal better in this respects.

Metals

Black body radiation

Black body: $5,67 \cdot 10^{-8} \text{ W/m}^2\text{K}^4$

2 cm diameter sphere ($12,6 \text{ cm}^2$), Black body, shiny copper and stainless steel

Radiation at RT 0,56 W, 0,02 W and 0,47 W

Heat transfer air 6-150, water 250-8000 $\text{J/m}^2\text{sK}$

Surface Material	Emissivity Coefficient - ϵ -
Alumina, Flame sprayed	0.8
Aluminum Commercial sheet	0.09
Aluminum Foil	0.04
Aluminum Commercial Sheet	0.09
Aluminum Heavily Oxidized	0.2 - 0.31
Aluminum Highly Polished	0.039 - 0.057
Aluminum Anodized	0.77
Aluminum Rough	0.07
Aluminum paint	0.27 - 0.67
Copper electroplated	0.03
Copper heated and covered with thick oxide layer	0.78
Copper Polished	0.023 - 0.052
Glass smooth	0.92 - 0.94
Glass, pyrex	0.85 - 0.95
Inconel X Oxidized	0.71
Mercury liquid	0.1
Nichrome wire, bright	0.65 - 0.79
Porcelain, glazed	0.92
Quartz glass	0.93
Silicon Carbide	0.83 - 0.96
Stainless Steel, weathered	0.85
Stainless Steel, polished	0.075
Stainless Steel, type 301	0.54 - 0.63
Titanium polished	0.19
Tungsten polished	0.04
Tungsten aged filament	0.032 - 0.35
Water	0.95 - 0.963

Metals

Welding, soldering

- Welding: TIG (Tungsten Inert Gas), pieces are melted together, no flux, no electrochemical corrosion. Al, Fe.
- Hard soldering: with 'silver' (no brazing). Beware of flux, check for cadmium
- Soft soldering: avoid if possible. Beware of flux (resin). Lead can evaporate. Tin can get tin-pest.

Plastics and elastomers

Teflon and

- Teflon: poly(tetrafluorethylene). Semi-elastomer. Cold flow. -200 - 260 C (HF, fluorophosgen evolution). Machining. Self lubricating. Out gassing. Not for O-ring, but for other sealings. Heat expansion differs from that of borosilicate glass!
- Teflon FEP: poly(tetrafluorethylene-hexafluorpropylene). Semi-transparent, thermoplastic, weldable. 200 C.
- Kel-F: poly(chlorotrifuorethylene). More rigid, less cold flow. 200 C.
- PFA: tetrafluorethylene-perfluorvinylether copolymer. 260 C.
- Tygon R-3603: PVC. -45 - 93 C (plastic at warm). 10^{-7} mbar.

Plastics and elastomers

Viton and

- Shore-hardness (50, 70, 90)
- Viton-A: hexafluoropropylene-1,1-difluoroethylene copolymer. Elastomer. -29 - 205 °C. Can absorb He (beware at leak hunt). O-ring, tubes. Not compatible with ammonia! Several composition. Less elastic than rubber.
- Kalrez: perfluoro-elastomer. Exceptional chemical compatibility. Up to 327 °C. Expensive. Chemraz, Simriz. .
- EPDM – ethylene-propylene(-x) copolymer to 120-150 °C
- FVQM – fluorsilicone, more compatible with chemical than silicone

Plastics and elastomers

Buthyl rubber, silicone rubber and

- Buthyl rubber: poly(isobuthylene) with some isoprene. Buna-N: nitril-rubber. Up to 80 C. Inexpensive.
- Silicone-rubber: -95 - 200 C, up to 260 C. No plasticizer. Permeable to air and water. Small mechanical resilience. DIY silicone can be OK in for vacuum after some curing period
- Epoxy-resin itself is vacuum compatible, without fillers are good vacuum adhesives.
- Silver chloride: About the same heat expansion and borosilicate, can be used to solder small leakage, p.e. on feed through.

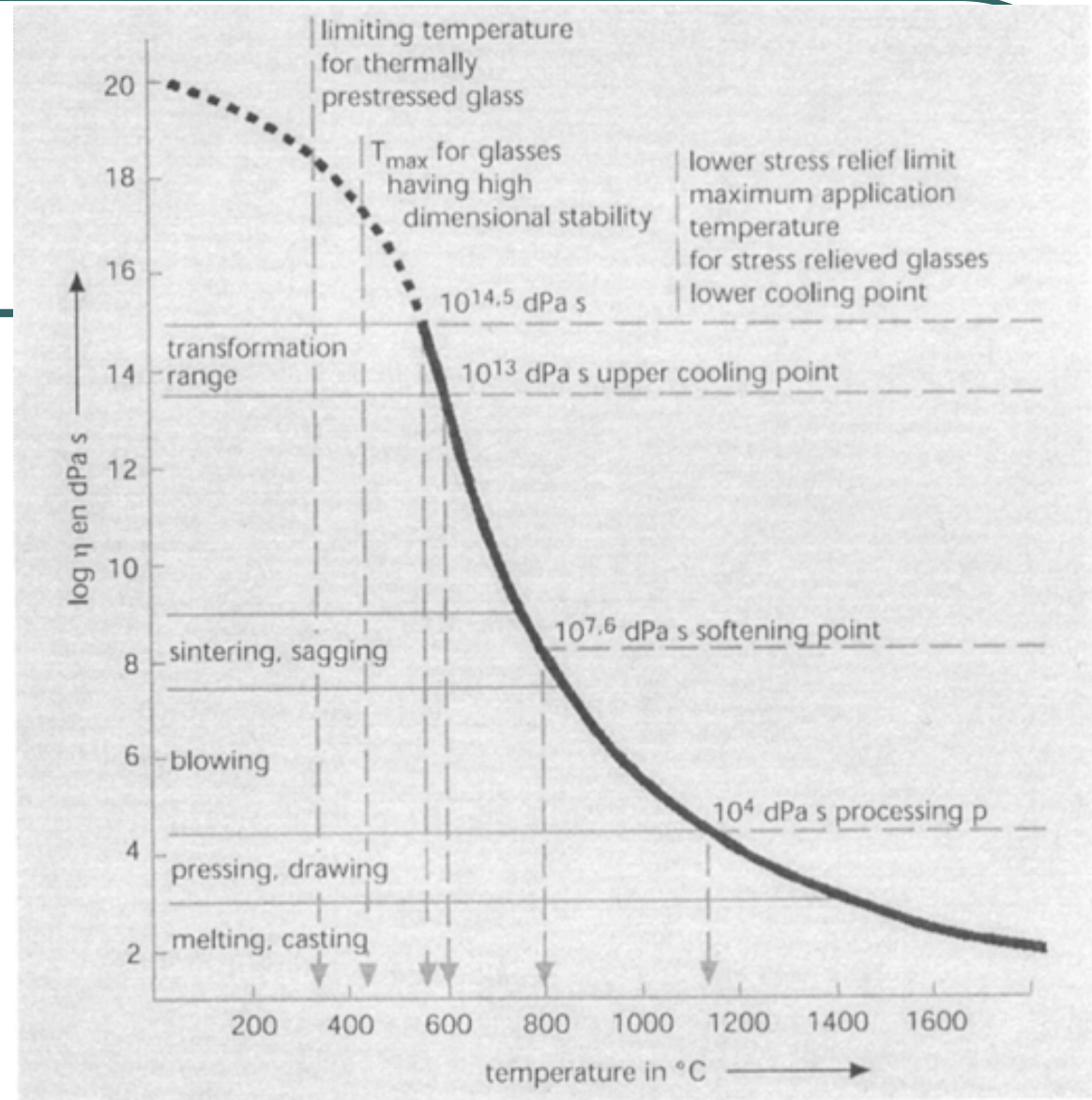
Plastics and elastomers

Others

- All plastics swell in solvents.
- Avoid unknown plastics. They can contain whatever.
- P.E. ordinary electric wire can be a temptation. But PVC insulation contains plasticizer, there is a lot of air between elemental wires.

Glass

- Borosilicate glass, 3.3 glass (ISO 3585). By thermal expansion: $3.3 \cdot 10^{-6} \text{ 1/K}$. 81% SiO_2 , 13% B_2O_3 , 4% $\text{Na}_2\text{O/K}_2\text{O}$, 2% Al_2O_3 . Pyrex, Simax, Duran, Kymax etc.
- Up to 500 C. (560 C annealing, 825 C softening, 1250 C working point). Down to 0 K.
- Can adsorb a lot of water.



Normal temperature dependence/viscosity curve of, for example, DURAN®; viscosity ranges of important processing techniques, position of fixed points of viscosity and various limiting temperatures.

Quartz

- Amorphous SiO_2 . Better UV and IR transparency than glass. Up to 1000 C, then starts to crystallize. $5.5 \cdot 10^{-7}$ 1/K. Melts, not softens! Hydrogen-oxygen torch.
- Do not alloy it to glass.
- Old pieces are still in use, can be recognized by rudimentary manufacturing
- Vycor: between borosilicate and quartz. 96% SiO_2 , 900 C, transparent.

Glass Transparency

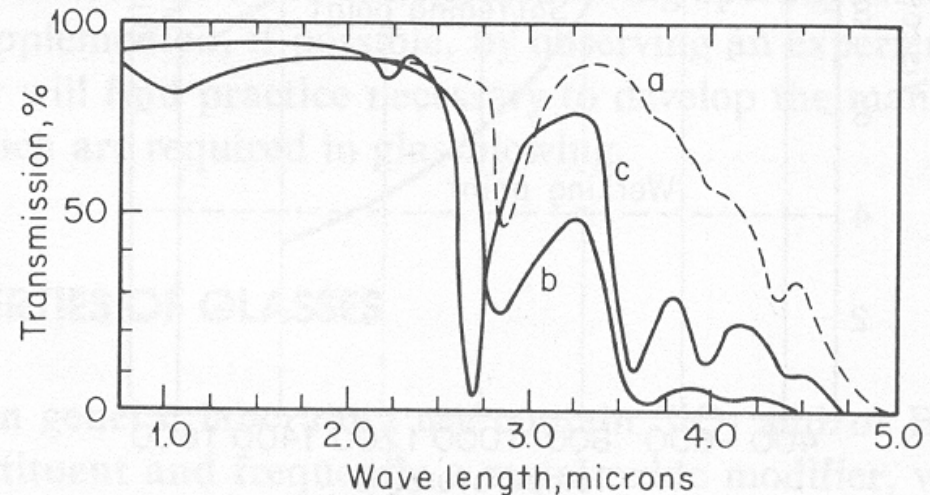
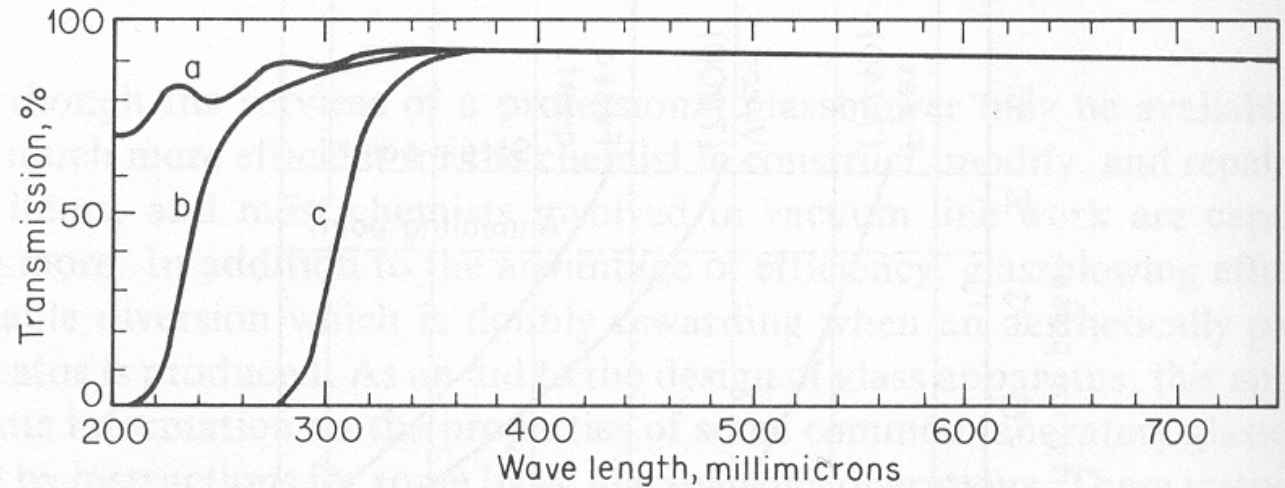


Fig. II.2. Transmission of Pyrex, Vycor, and fused quartz. Transmission curves for 2-mm-thick samples of (a) fused quartz (GE type 102), (b) Vycor (Corning 7910—note that this is a specially controlled grade; the more common 7900 has an ultraviolet cutoff similar to 7740), and (c) Pyrex (Corning 7740). (Adapted from data supplied by Corning Glass Works and General Electric Co.) Certain glasses and types of fused quartz are available which lack the infrared absorption at about 2.7 microns.

Ceramics

Aluminum oxide (sapphire), BN, SiC

- Refractory, good insulators, good phys-chem compatibility. Brittle.
- Sapphire UV and IR transparent, no scratches.
- Can be porous.
- Can be silver soldered

Oils, greases

„Vacuum grease”

- Glass joints. Mechanical lubrication of O-rings
- Silicone grease: $<10^{-6}$ mbar. Solvents can dissolve, alkaline materials decompose. -20 - 100 C.
- Mineral oil (Apiezon series, L, M, N, T): 10^{-7} - 10^{-10} mbar, 30 (80) C. There are waxes for semipermanent use (W, max. 80 C, melts at 100 C).
- Kel-F 200: perfluorinated wax. 40 (90) C.

Oils, greases

Rota-oil, turbó-oil

- Rota-oil: refined mineral oil (ester). Lubricates, seals and cools. Exchange on suspicion. Trap it (molecular sieve X-13), can contaminate HV. There are fluorinated versions.
- Turbo-oil: even more refined. Lubricates and cools.

Oils, greases

Diffusion pump oils

- Mercury is the past.
 10^{-3} mbar

- Esthers, silicones, polyphenyl ethers, alkanes, fluorinated versions: there is a selection :-)

SOME DIFFUSION PUMP FLUIDS

Trade Name	Chemical Name	MW (ave)	P_v at 25°C (Pa)	Viscosity at 25°C (mm ² /s)	Boiler Temp. at 100 Pa (°C)
Convoil®-20	Hydrocarbon	400	5×10^{-5}	80	210
Octoil-S®	Bis (2-ethyl-hexyl) sebacate	427	3×10^{-6}	18.2	220
Invoil®	Diocetylphthalate	390	3×10^{-5}	51	200
Dow Corning®-704	Tetraphenyl-tetra methyl trisiloxane	484	3×10^{-6}	38	220
Dow Corning®-705	Pentaphenyl-tri methyl trisiloxane	546	4×10^{-8}	175	250
Santovac 5®	Mixed 5-ring polyphenylether	447	6×10^{-8}	2400	275
Fomblin® Y VAC 25/9	Perfluoropoly-ether	3400	9×10^{-7}	190	230

Oils, () Diffus

Characteristics of Diffusion Pump Fluids

- (1) Hydrocarbon Oils
 - (a) low cost
 - (b) decompose on air exposure
 - (c) o.k. for gauges and ion sources
 - (2) Silicone Compounds
 - (a) moderate cost
 - (b) good on air exposure
 - (c) bad for gauges and ion sources
 - (3) Polyphenyl Ether
 - (a) high cost
 - (b) good on air exposure
 - (c) o.k. for gauges and ion sources
 - (4) Fatty Esters
 - (a) moderate cost
 - (b) poor on air exposure
 - (c) o.k. for gauges and ion sources
 - (5) Halogenated Compounds
 - (a) high cost
 - (b) oxygen compatible
 - (c) may be decomposed by Lewis acids
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Brands of Diffusion Pump Fluid

- (1) Hydrocarbon Oils
 - (a) Apiezon A, B, C
 - (b) Litton oil
 - (c) Convoil-20
 - (2) Silicone Compounds
 - (a) DC-704
 - (b) DC-705
 - (c) Invoil 940
 - (3) Polyphenyl Ether
 - (a) Santovac 5
 - (b) Convalex 10
 - (4) Fatty Esters
 - (a) Octoil and Octoil-S
 - (b) Butyl Phthalate
 - (c) Amoil and Amoil-S
 - (d) Invoils
 - (5) Fluoroether Polymers
 - (a) Krytox
 - (b) Fomblin
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Other stuff

Contaminating, cleaning

- Fingerprint is an enemy
- Machining fluids
- Introduced materials: as you please. Beware of reactions.
- Acetone can have evaporation residue. 96 % ethanol, chloroform-ethanol 1:1 mixture.
- Paper, gauze, gloves.
- Ultrasonic bath.
- Cover against dust
- (Sources)