Technical description of photoelectron spectrometer Escalab 250Xi

Resource center «Physical Methods of Surface Investigations»

2014

Table of contents

Common description

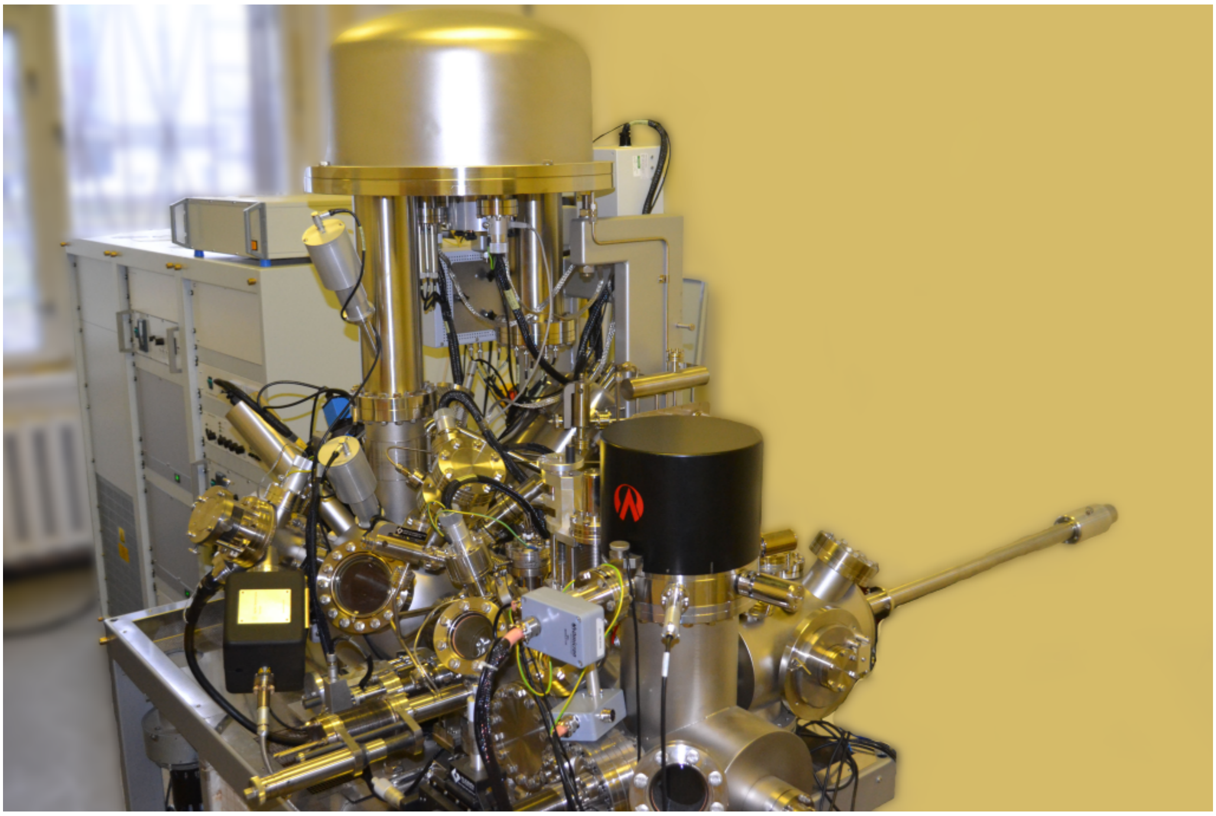


Figure 1: Common view of experimental station.

Experimental station Escalab 250Xi (see fig. 1), designed by Thermo Fisher Scientific[1], is designated for realization of elemental and chemical analysis of surface of samples. Next methods are realized in the station:

1. X-ray photoelectron spectroscopy (XPS) and X-Ray Photoelectron Imaging (XPi)
2. Ultraviolet photoelectron spectroscopy (UPS)
3. Auger electron spectroscopy (AES) and Scanning Auger Electron Mapping (SAM)
4. Scanning Electron Microscopy (SEM)
5. Ion Scattering Spectroscopy (ISS)
6. Electron Energy Loss Spectroscopy (EELS)
7. Raster ion etching
8. Low-energy Electron Diffraction (LEED)
9. Charge compensation system for insulating samples
10. Sample heating in controlled gas environment

[1]see http://www.thermoscientific.com/en/product/escalab-250xi-x-ray-photoelectron-spectrometer-xps-microprobe.html and http://xpssimplified.com/escalab_250xi.php

Control of gas lines, pump-down systems and process of measurement is realized in special software Avantage[2] (see fig. 2). Samples are assembled to standard sample holder kit Thermo Fisher Scientific SKIT20 (see section Requirements for samples. Sample holders.

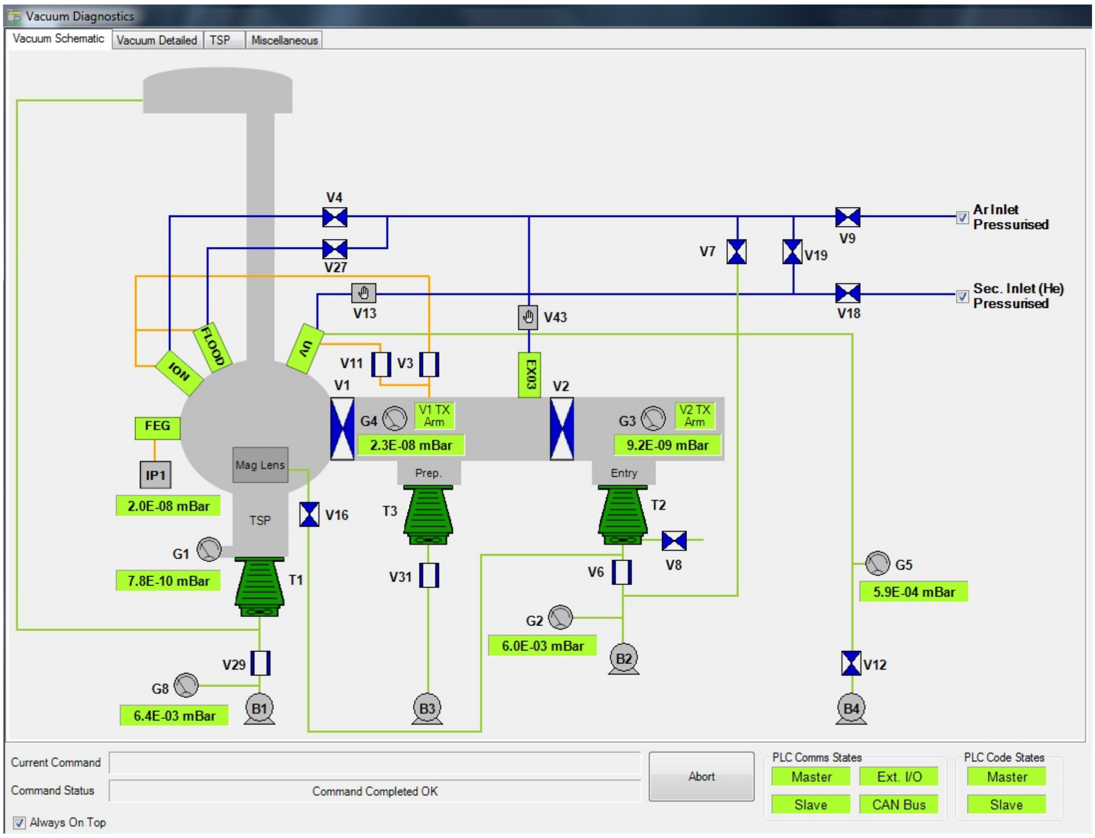


Figure 2: View of window of Avantage software.

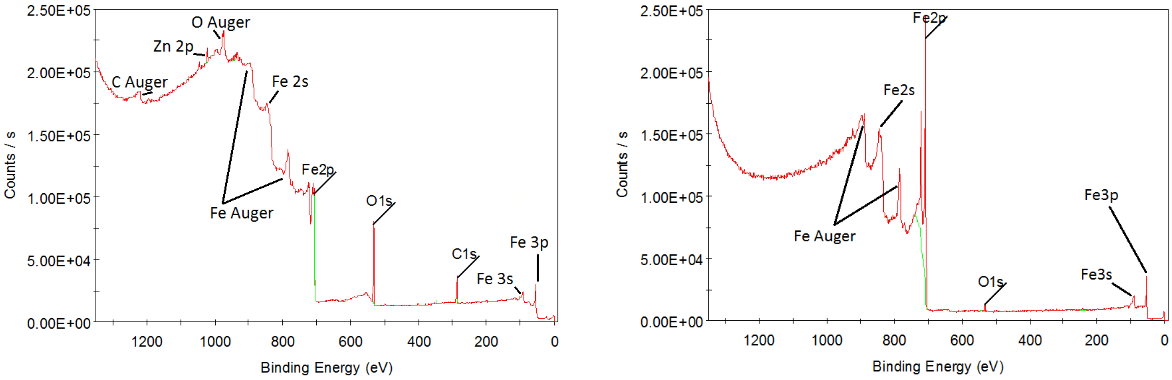
Possible objects of investigations:

1. monocrystals
2. polycrystalls
3. thin films
4. powders

Available information about objects:

1. quantitative analysis
2. chemical shifts of core levels

[2]see http://xpssimplified.com/avantage_data_system.php



(a) (b)

Figure 3: XPS spectra of steel 09G2S: (a) before etching, (b) after ion etching

Table 1: Determination of chemichal composition of steel 09G2S

	Elemental composition of the sample			
	Initial state		State after etching	
Element	**Energy (eV)**	**Atomic fraction (%)**	**Energy (eV)**	**Atomic fraction (%)**
Fe2p	707.01	1.91	707.45	92.22
O1s	531.28	67.89	531.19	7.78
C1s	285.87	30.20		

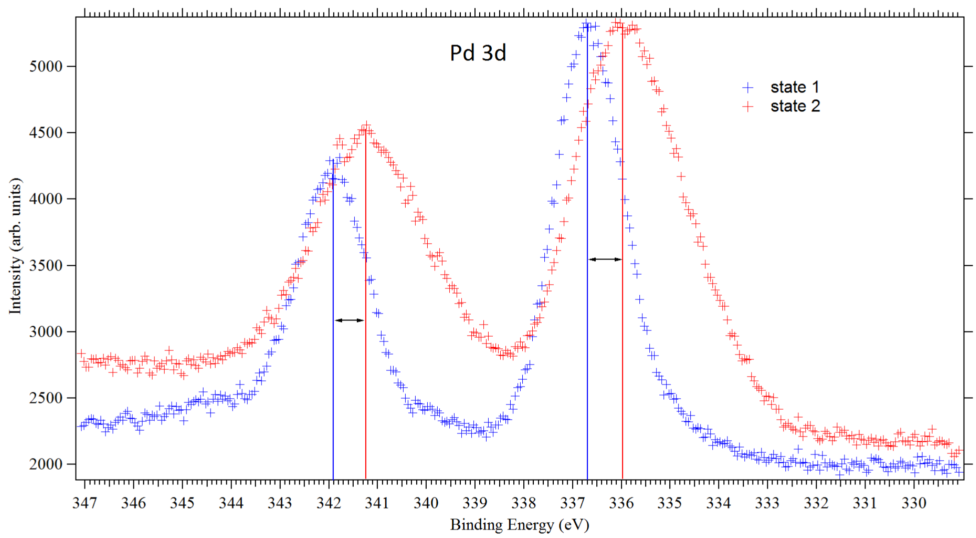
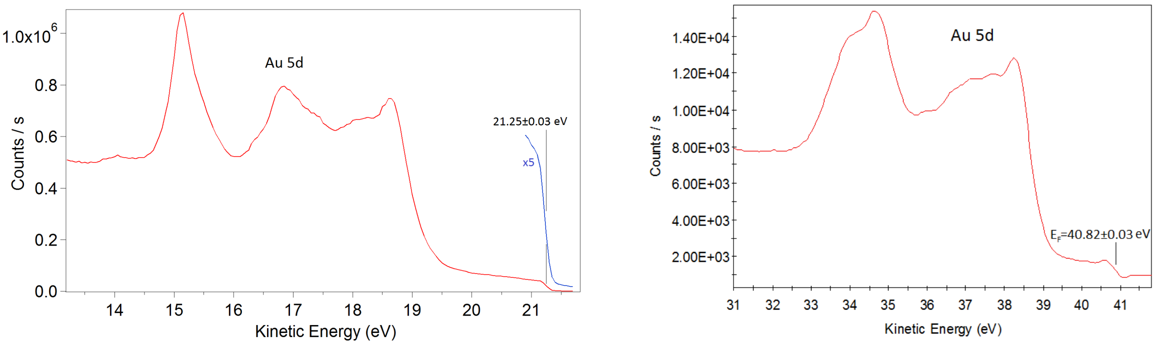


Figure 4: Core levels of palladium being in different states, shift - 0.7 eV.

3. electronic structure of valence band



(a) (b)

Figure 5: Photoelectron spectra of valence band of polycrystalline gold: (a) HeI (21.2 eV), (b) HeII (40.8 eV)

4. depth profile of elements in near-surface area

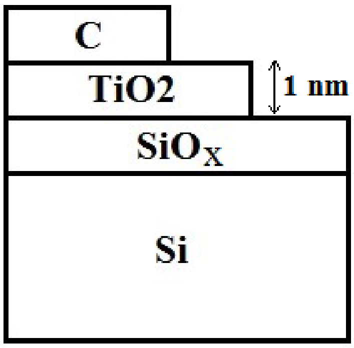


Figure 6: Layer-by-layer scheme TiO_2/Si. Data about element distribution and value of film thicknesses are obtained from analysis of spectra, measured for different emission angles relative to normal of sample surface

5. map of chemical states of elements on surface

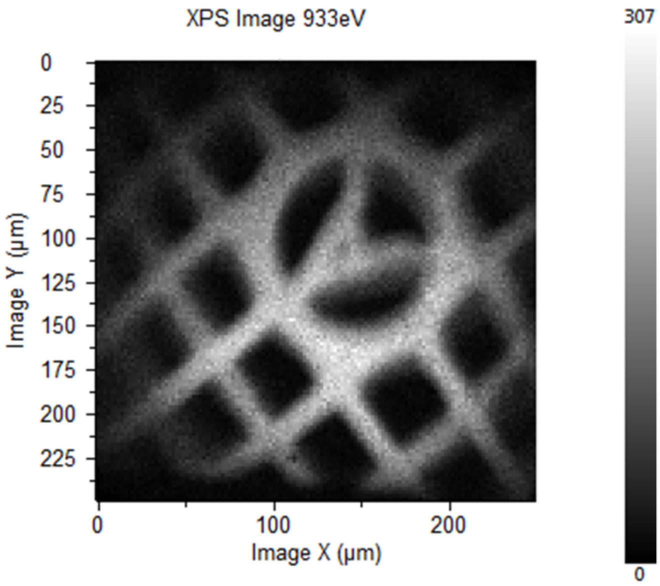


Figure 7: map of copper on surface of test sample

6. information about crystallographic structure and orientation in the surface Brillouin zone

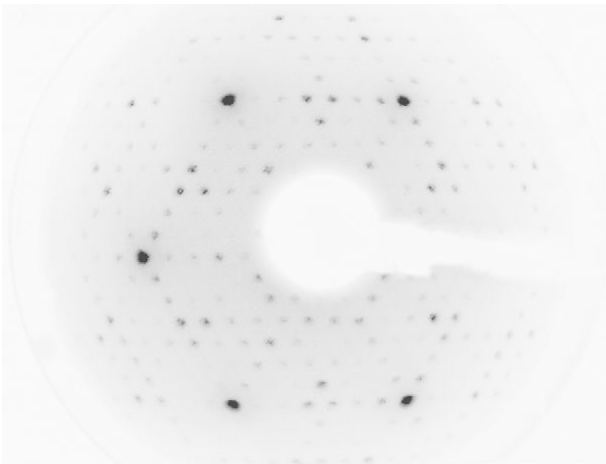


Figure 8: LEED image of Si(111)7×7. Beam energy - 50 eV.

Analytical chamber

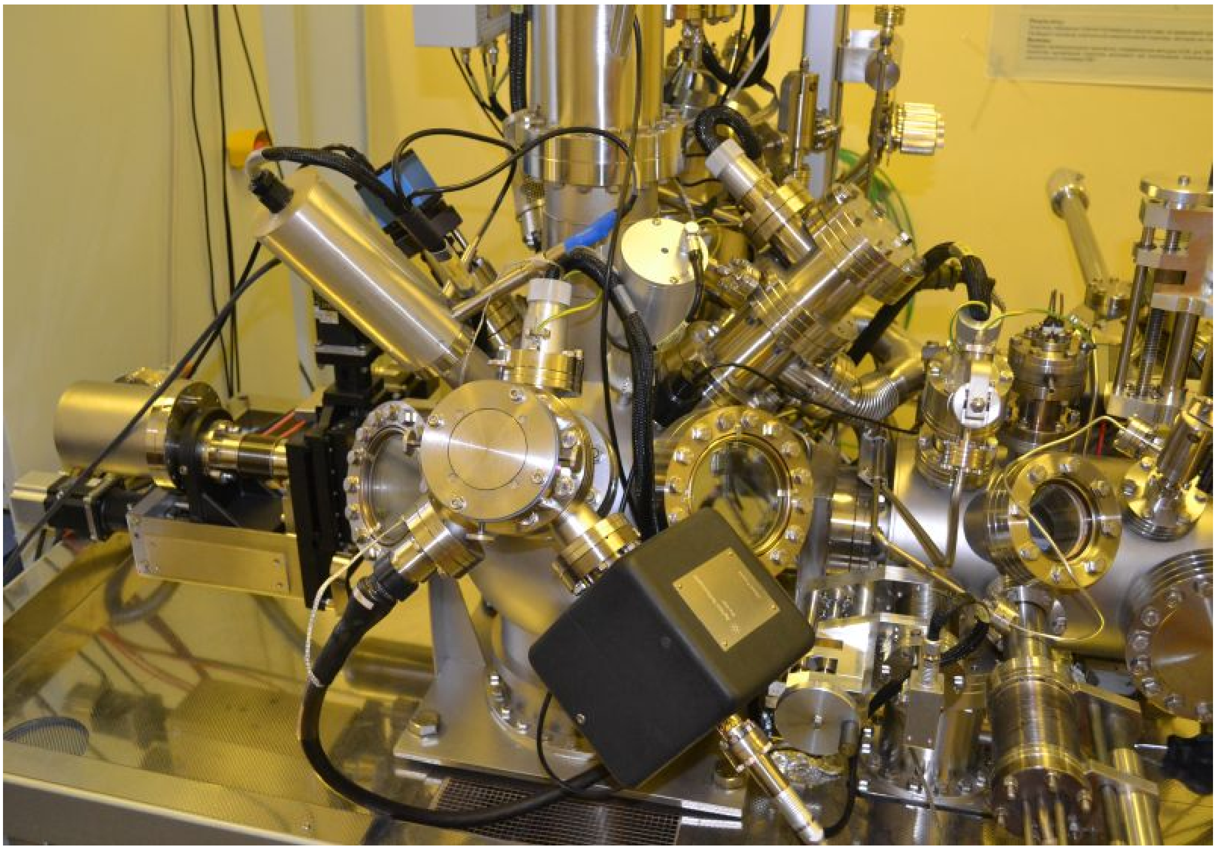


Figure 9: Analytical chamber.

Experimental station consists of 3 chambers, which have independent pump-down systems: analytical chamber (see fig. 9), preparation chamber (see fig. 11) and load lock (see fig. 12). Base pressure in analytical chamber -1 $\cdot 10^{-10}$ mbar.

Analytical chamber is equiped by the following devices (see fig. 10):

1. X-ray source Al-K_α (1486.6 eV, with monochromator), spot size is adjusted from 200 microns to 900 microns. Minimal analysis area is less than 20 μm

2. ultraviolet source (HeI (21.2 eV), HeII (40.8 eV)) with differential pump-down system for investigations of valence band with angle resolution less than 1.5°[3]

3. system for XPS imaging with spatial resolution less than 3 microns

4. raster ion gun EX06 (energy of ions from 0.2 to 4 keV)

5. electron source build-in to lenses of analyser for neutralisation of positive charge and source of positive argon ions for neutralisation of negative charge (possibility of measuring of insulators)

6. hemispherical analyzer with 2 detectors: multi-channel detector, based on channel electron multipliers with wide dynamic rang and 2D-detector based on twin microchannel plate and coordinate detector

[3]see http://www.datacompscientific.com/TVGS/UVL_AN31058_DS.pdf

7. system of magnetic immersion lenses for increasing of sensivity (intensity) with saving of spatial resolution

8. raster ion gun based on effect of field emission FEG1000 with spot size less than 95 nm at current 5 nA and energy 10 keV[4], Secondary Electron Detection (SED) system

9. digital camera The Imaging Source DFK 41AF02[5]

10. 5-axis manipulator, which allows to hold the sample, save several points of measurements and restore position after moving to preparation chamber

11. system of heating of sample at manupulator (up to 1000 K)

Figure 10: Devices in analytical chamber.

Preparation chamber

[4]see http://www.datacompscientific.com/TVGS/FEG1000_AN31059_DS.pdf

[5]see http://www.theimagingsource.com/en_US/products/cameras/firewire-ccd-color/dfk41af02/

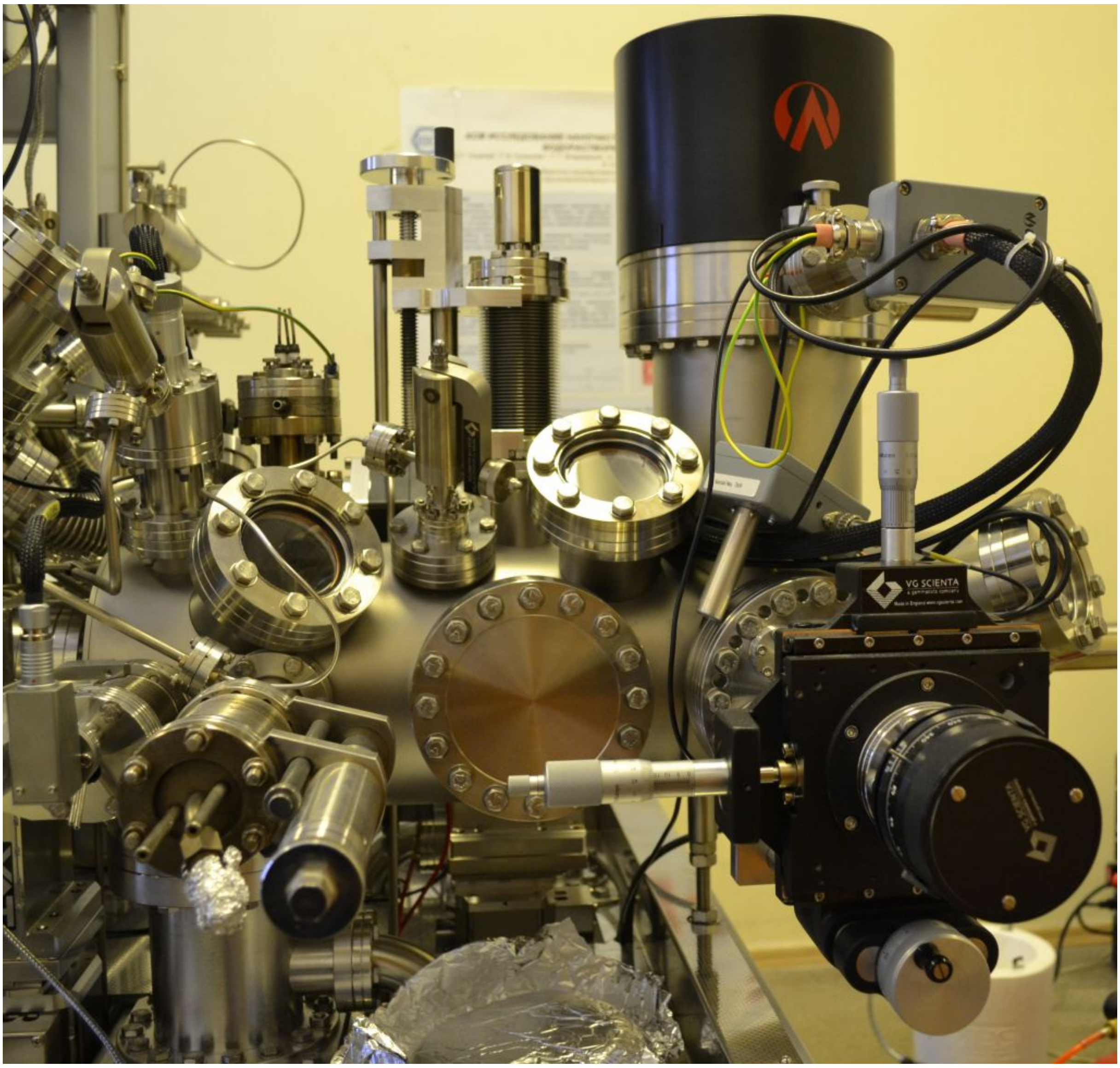


Figure 11: Preparation chamber.

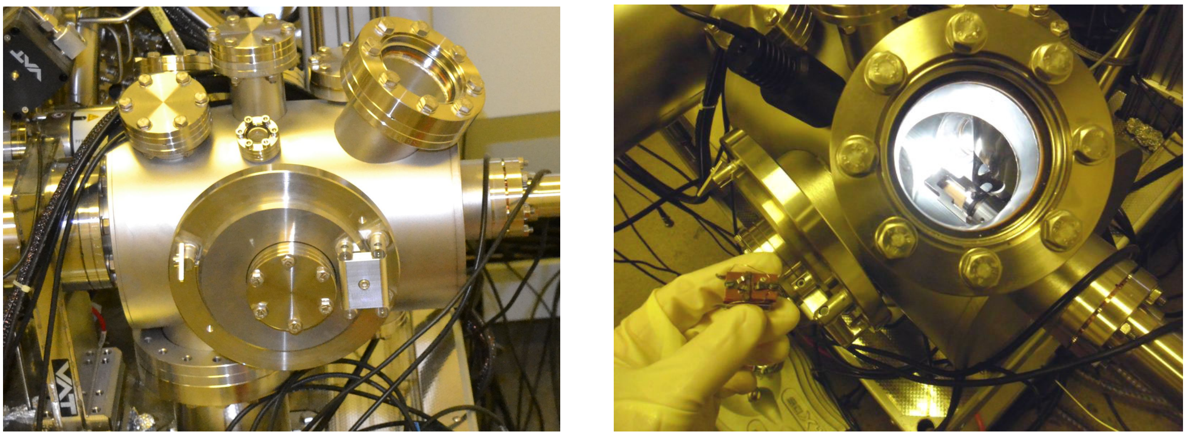
Preparation chamber chamber is equiped by the following devices:

1. ion gun EX03[6]
2. diffractometer Omicron SPECTALEED with Auger electron optics[7]
3. stage for heating of samples (up to 1000 K)
4. gas inlet system (it is possible to measure in gas environment with pressure $1 \cdot 10^{-8}$ - $1 \cdot 10^{-5}$ mbar)
5. storage for 3 sample holders

[6] see http://www.datacompscientific.com/TVGS/EX03_AN31062DS.pdf

[7] see http://www.omicron.de/en/products/spectaleed-/instrument-concept

Load-lock



(a)

(b)

Figure 12: Load-lock: (a) common view, (b) loading of sample holder to load lock

Load-lock is a part of system, where sample holders are inserted from atmosphere and are taked out after measurements. Loading is realized through the door, closed with viton o-ring (see fig. 12(b). It is possible to take out from experimental station one sample holder, arranged at transfer probe, and install another one (or origin sample holder after change of samples) to its place during one cycle. Load-lock is not baked out after loading of samples, but pump-down system allows to get pressure about $1 \cdot 10^{-9}$ mbar.

Requirements for samples. Sample holders.

Samples are installed to one of sample holders from kit Thermo Fisher Scientific SKIT20.

It is possible to measure solid-state samples, which don't break ultrahigh vacuum conditions during measurements. Permissible length and width of samples vary from used sample holder, maximal height with using standard sample holder (Sample Carrier Block) equals 5 mm, maximal height with using sample holder for heating (Heated Sample Assembly) - 3 mm. It is allowed to install several samples at one sample holder. If samples are powders, users should preliminary install (press in) samples to appropriate substrate, for example, soft metal (copper, gold) or vacuum sticky tape. It is allowed to compress powders to tablets. If samples are conductive, substrates should be conductive too. It is allowed to measure dried suspension, which are carried on hard plain surface, for example, silicon. Preparation of samples and substrates (clipping, infliction of substances, drying etc.) is carried out by users, direct installing to sample holder - by stuff of resource center.

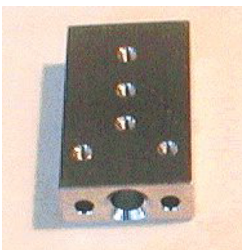


Figure 13: Sample Carrier Block.

The Sample Carrier Block (see fig. 13) is a 25mm long by 14mm wide stainless steel by 6mm block. To attach samples to this Holder, the sample can either be attached directly to the Sample Carrier Block by using the M2 screws (see fig. 14) or clipped down using the molybdenum sample retainers with M2 screws. Alternatively for large samples tape or silver dag can be used (see fig. 15).

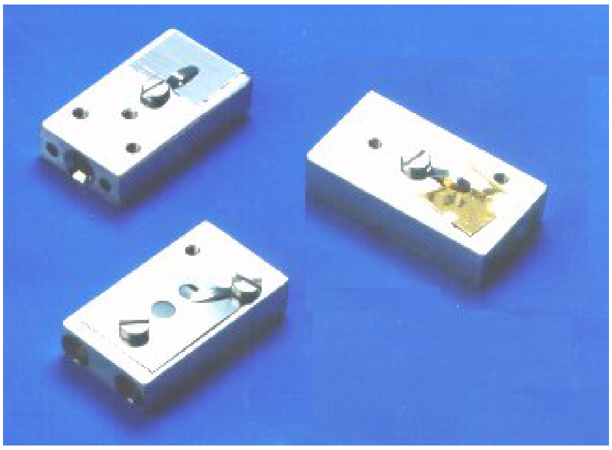


Figure 14: Sample Carrier Blocks loaded using retainers.

Figure 15: Sample Carrier Block loaded using silver DAG or double sided tape.

The Multi-Sample Assembly comprises of a 50mm long by 20mm wide by 26SWG stainless steel plate mounted on a stainless steel Sample Carrier Block. To attach samples to this Holder, sample clips can be used either the type. Alternatively samples can be attached to this holder, using the 8 holes down the sided and clipped down by the M1.6 screws

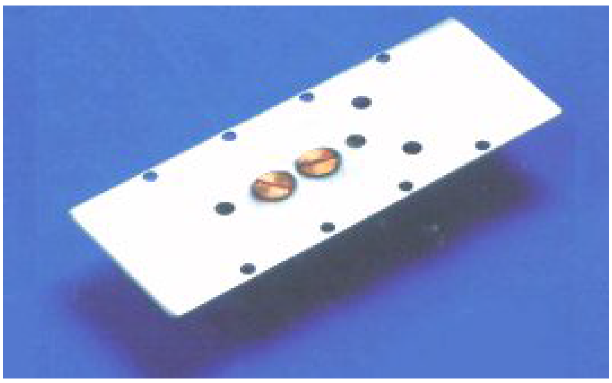


Figure 16: 50mm Multi-sample Assembly.

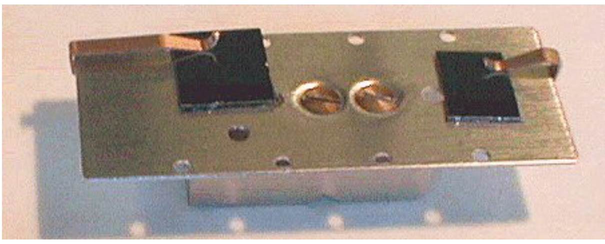


Figure 17: 50 mm Multi-Sample Assembly loaded using clips.

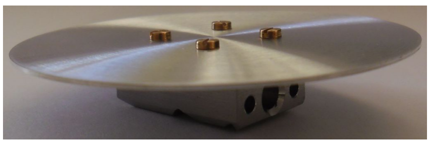
Figure 18: 50mm Multi-Sample Assembly loaded using sample retainers.

Improved depth resolution may be obtained for some materials if the sample is rotated during sputtering to reduce topography formation. Materials that tend to show improved depth resolution with rotation are polycrystalline metals on flat substrates, e.g. aluminium on silicon.

Rotation can also be useful when profiling rough samples, as any shadowing of the ion beam by roughness on the sample is minimized.

Rotation of the sample during sputtering is possible due to 5-axis manipulator and a rotatable sample holder, which consists of block, gear and metallic disc (see Fig. 19).

(a)



(b)

Figure 19: Azimuthal Rotation Holder Assembly: (a) before installing a disc, (b) after installing a disc

Special sample holder (see Fig. 20) allows to heat the sample with temperature up to 600 K and cool down the sample by use of liquid nytrogen to temperature 170 K. System contains a thermocouple for feedback control.

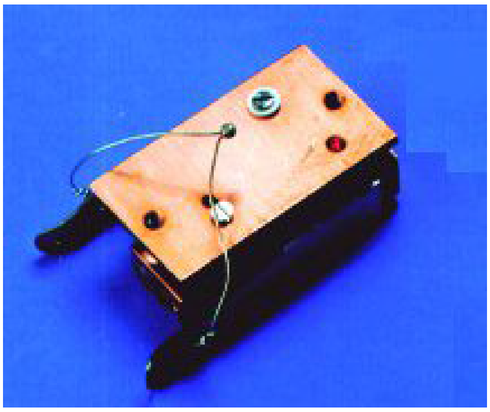


Figure 20: 600 deg K. Heated Sample Assembly.

Sample holder for intensive heating (see Fig. 21) allows to heat the sample with temperatures up to 1000 K.

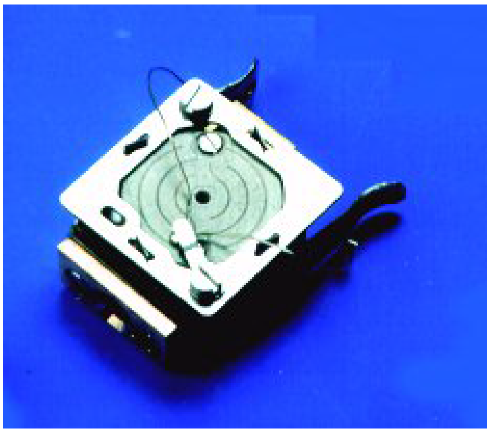


Figure 21: 1000 K Heated Sample Assembly.

Appendix A: main drawings of experimental station

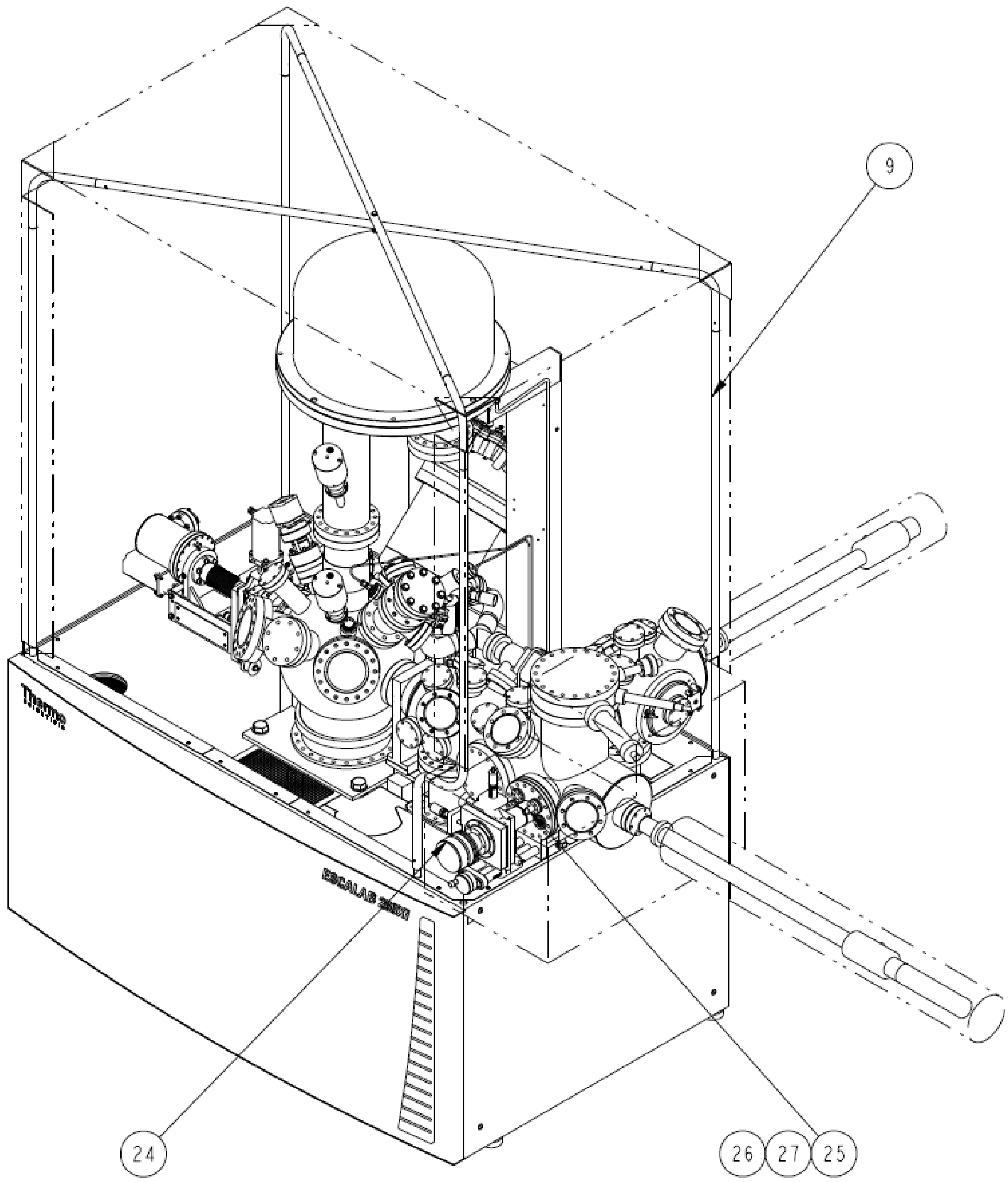


Figure 22: Drawing of experimental station.

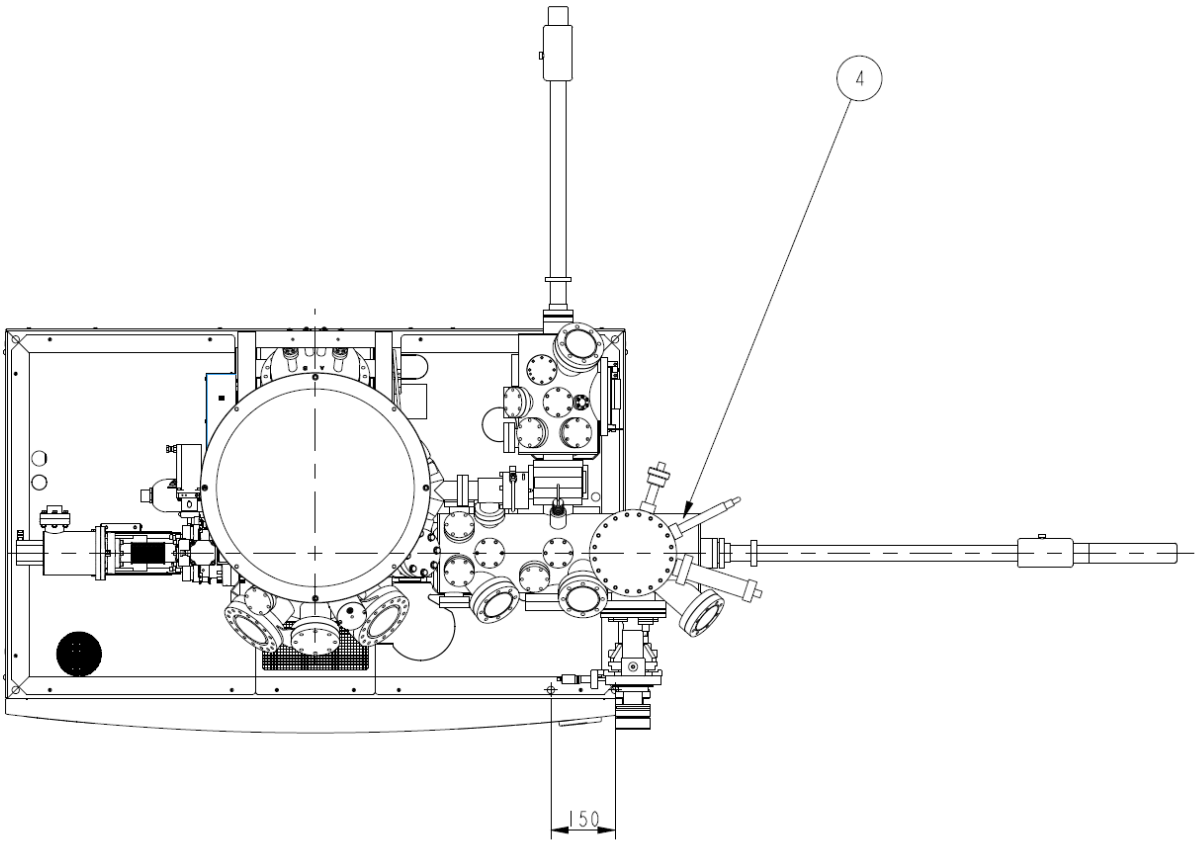


Figure 23: Drawing of experimental station. Top view. On the left: analytical chamber, on the bottom right - preparation chamber. on the top right - load lock chamber.

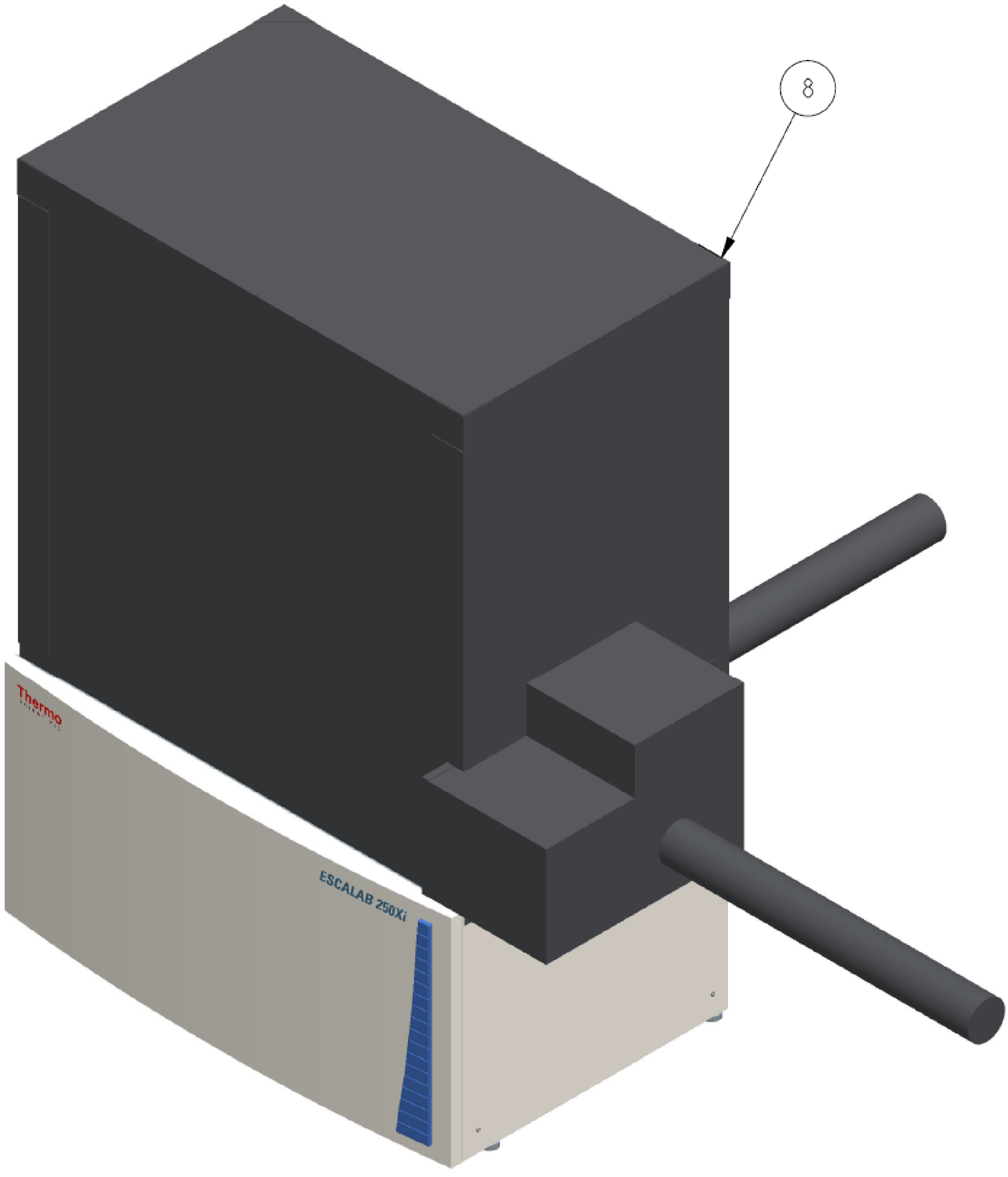


Figure 24: Common view of experimental station with bakeout tent.

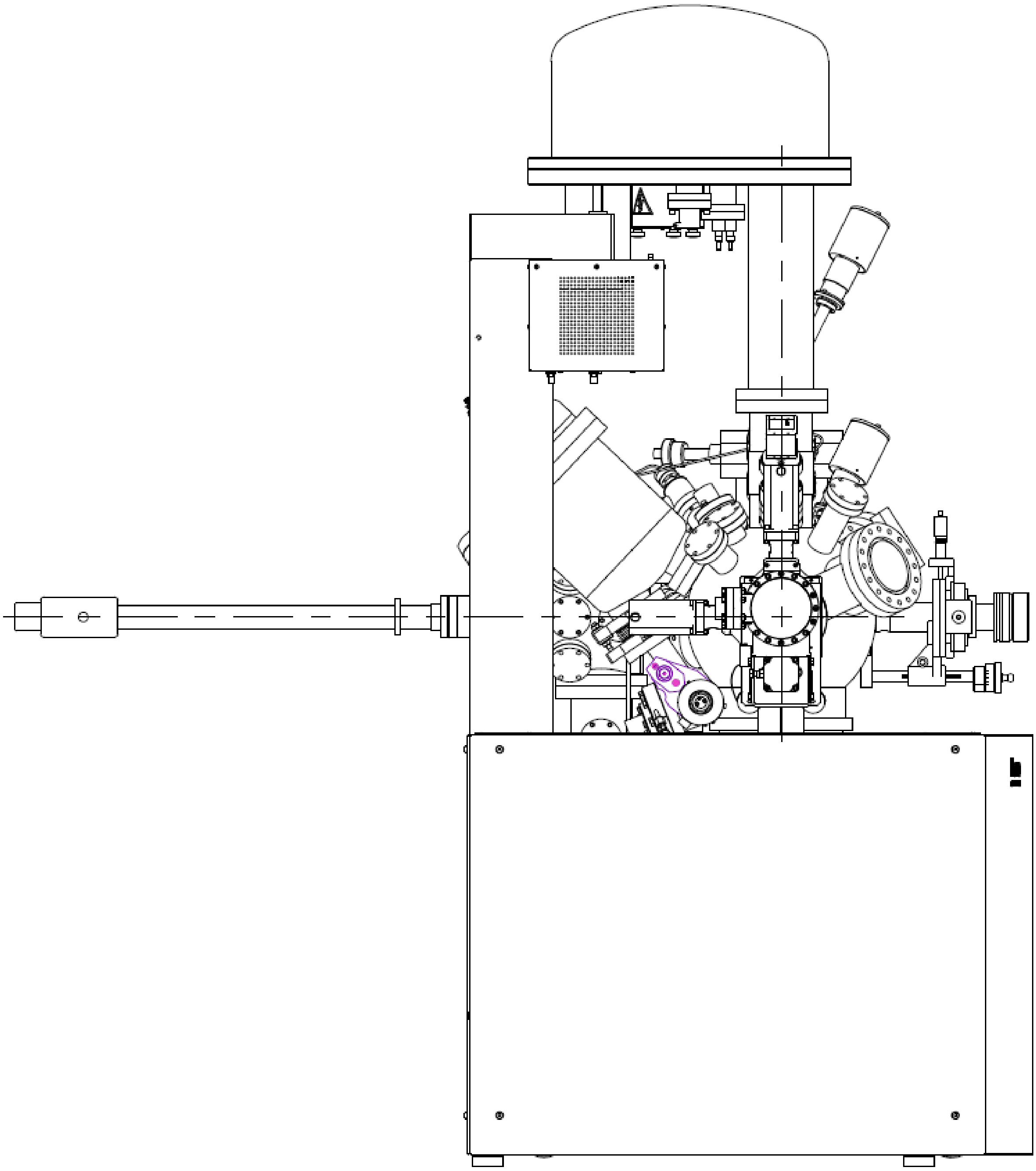


Figure 25: Drawing of experimental station. View from the analytical chamber.

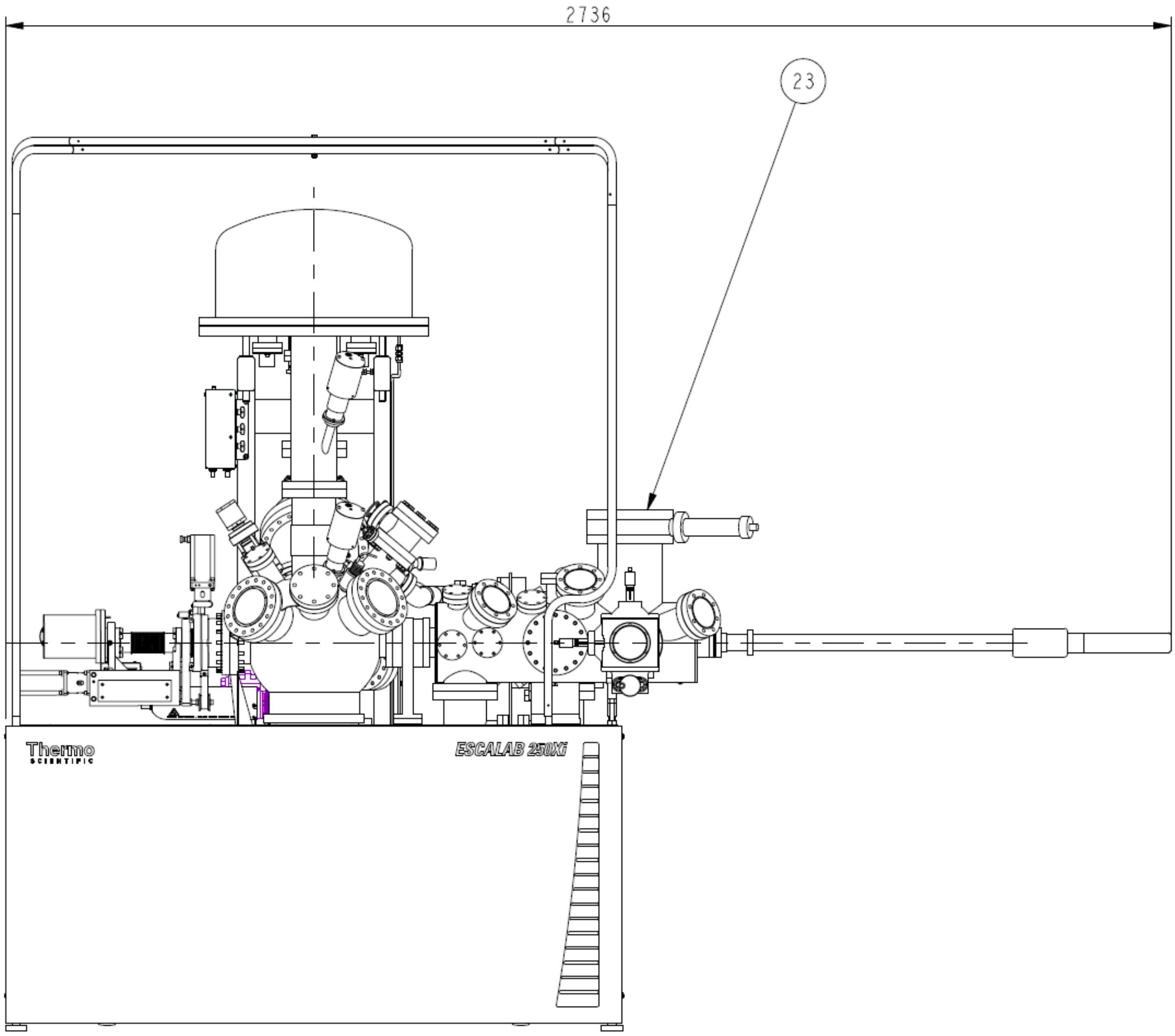


Figure 26: Drawing of experimental station. Front view. On the center - analytical chamber, on the right - preparation chamber.

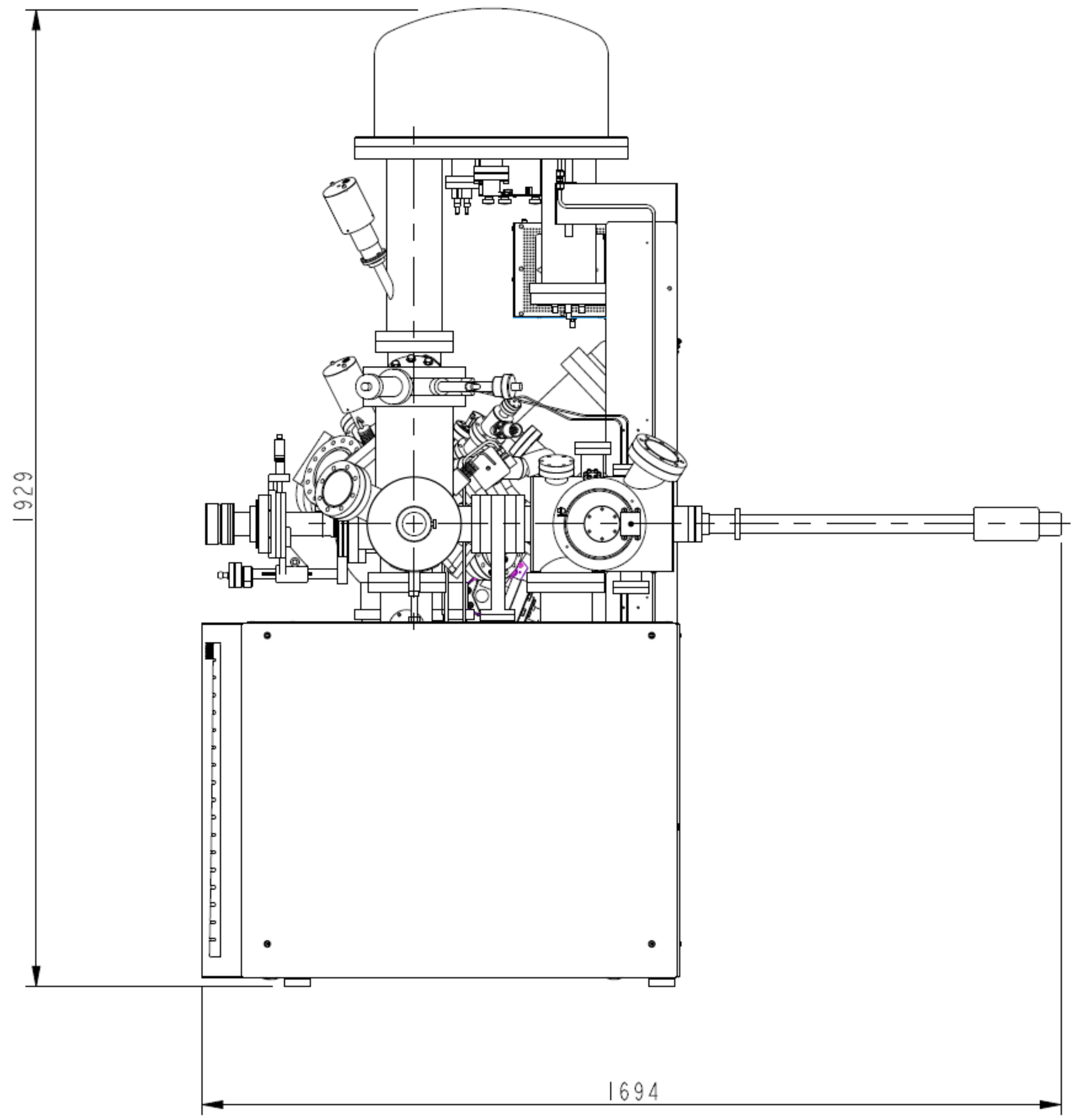


Figure 27: Drawing of experimental station. View from the preparation chamber (right view). On the left - preparation chamber, on the right - load-lock chamber.

Table 2: Flanges and equpment of experimental station Escalab 250Xi

No	Description
4	UHV preparation chamber
8	bakeout tent
9	tent frame
23	LEED/Auger Omicron SPECTALEED
24	sample holder ZSHI[8] and XL09 sample receiver
25	coaxial feedthrough
26	Miniax translator ZXYZ1015[9]
27	rotary motion drive RD2[10]

[8] see https://www.vgscienta.com/_resources/File/Catalogue_Sections/Manipulation/Manipulation-SampleHolders.pdf

[9] see http://www.vgscienta.com/_resources/File/Catalogue_Sections/Manipulation/Manipulation-Miniax.pdf

[10] see http://www.vgscienta.com/_resources/File/Catalogue_Sections/VGScienta_Rotary_Drives.pdf

Appendix B: drawings of analytical chamber

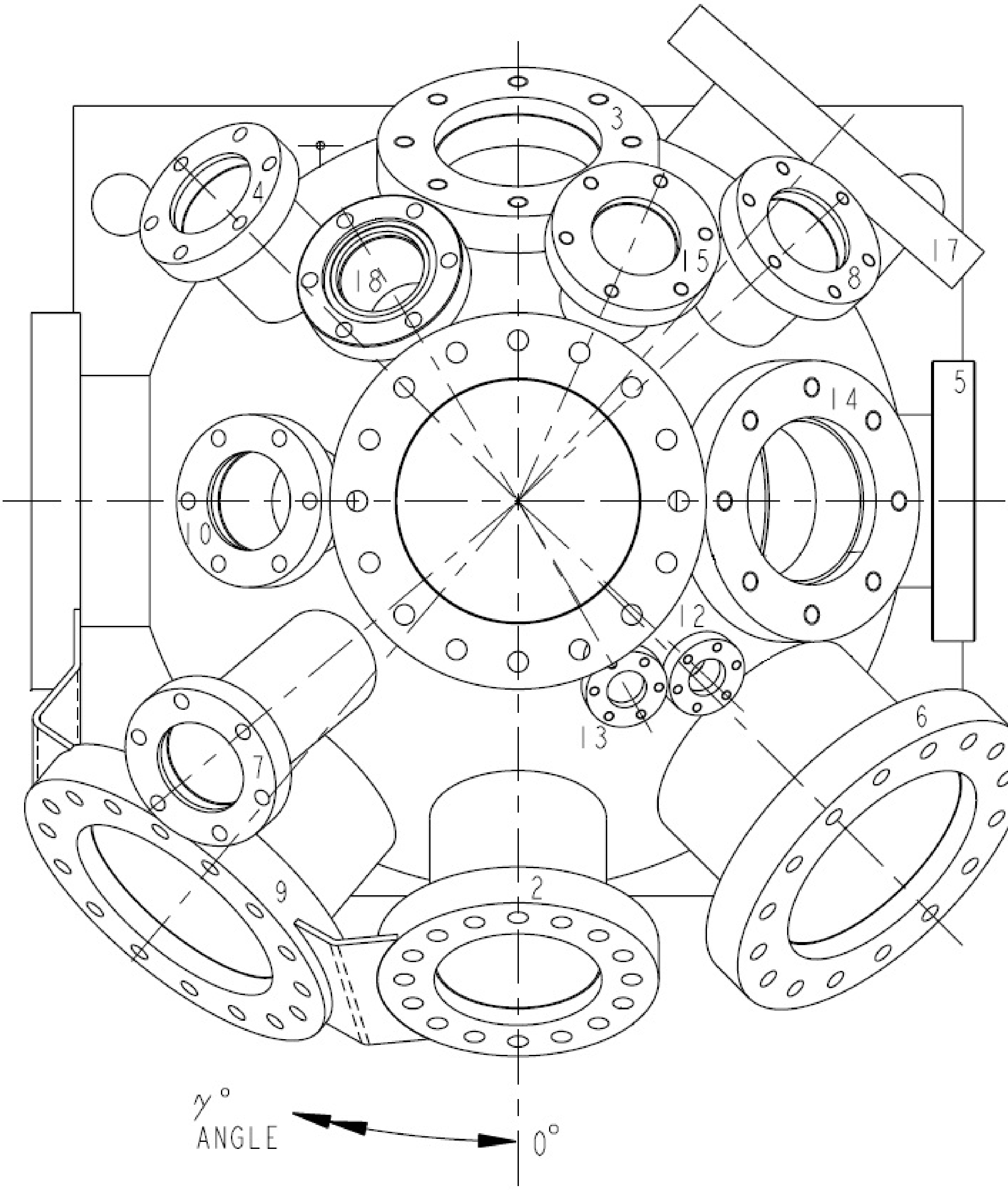


Figure 28: Drawing of analytical chamber. Top view (on the right - preparation chamber).

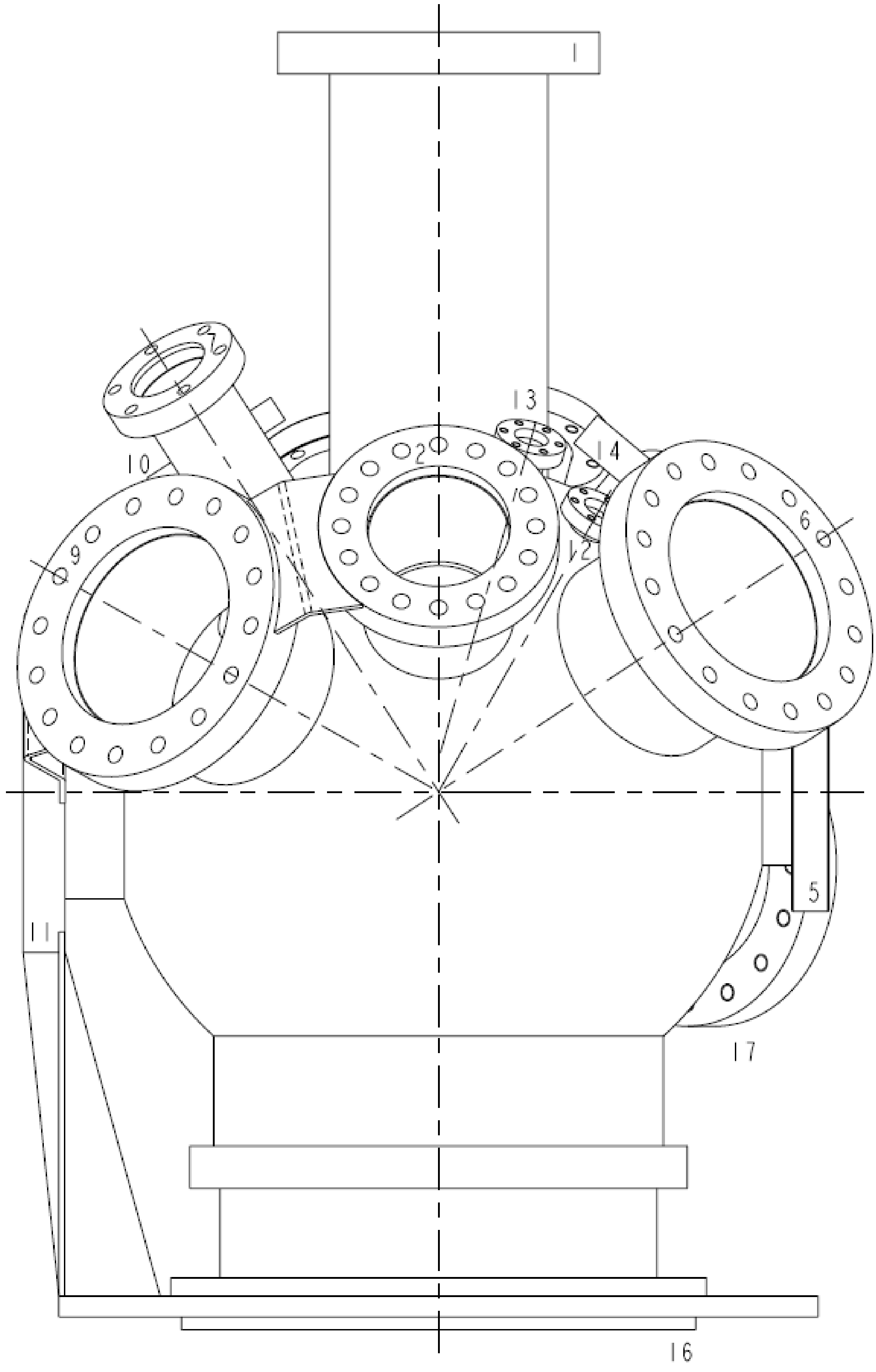


Figure 29: Drawing of analytical chamber. Front view (on the right - preparation chamber).

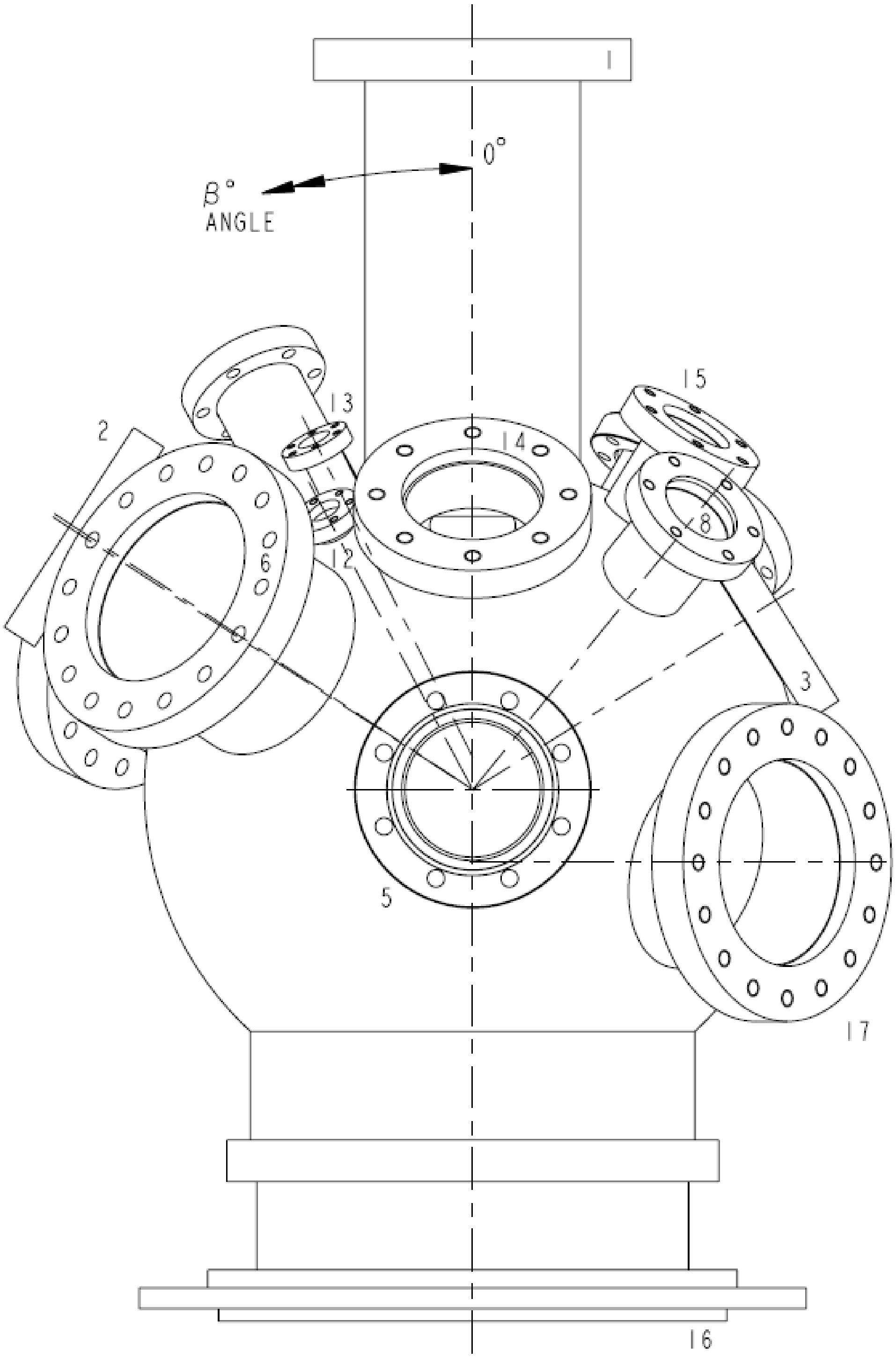


Figure 30: Drawing of analytical chamber. View from the preparation chamber (right view).

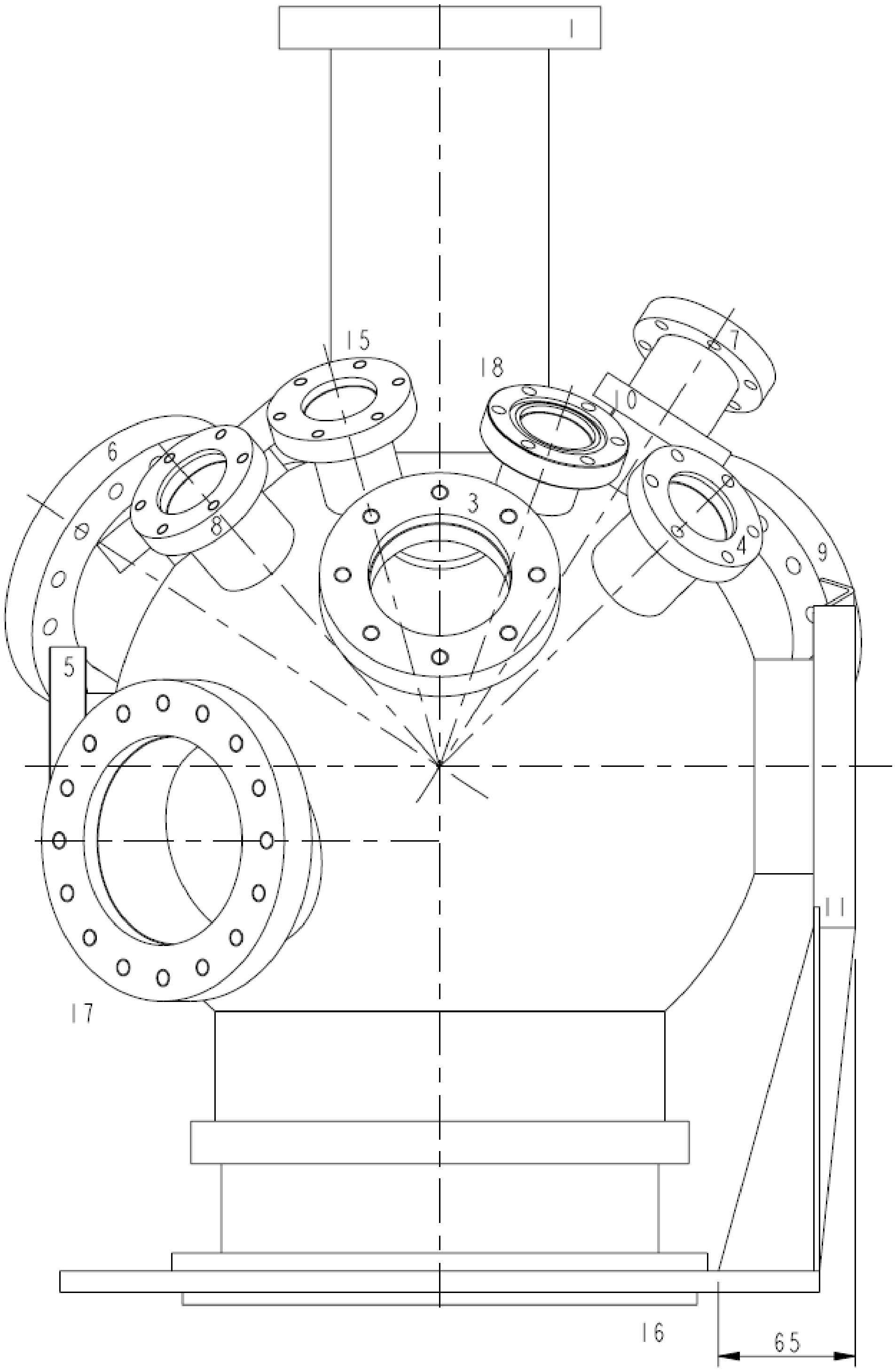


Figure 31: Drawing of analytical chamber. Back view (on the left - preparation chamber).

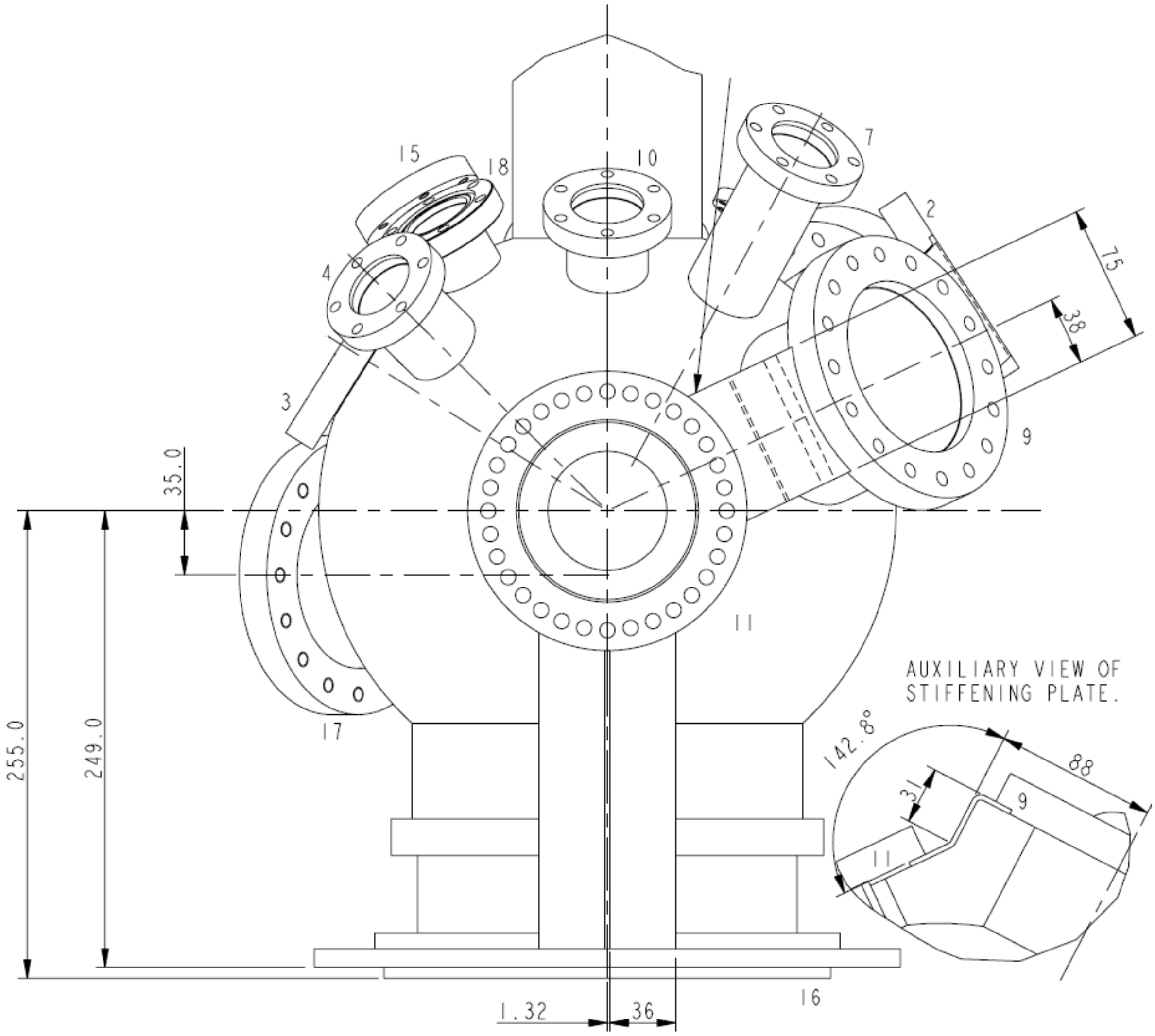


Figure 32: Drawing of analytical chamber. Left view.

Table 3: Flanges and equpment of analytical chamber

No	Tube size	Flange type	angle β	angle γ	Designation
1	-	DN100	0	0	lens assembly
2	71.65mm OD x 2.5mm	DN63	57	0	raster electron gun FEG1000
3	3/4” O/D 16 SWG	DN63	58	180	monochromator and X-Ray source assembly (XR6)
4	3/4” O/D 16 SWG	DN35	55	135	vacuum gauge
5	3/4” O/D 2.5mm	DN63	90	270	preparation chamber
6	O/D” O/D 16 SWG	DN100	65	315	viewport
7	3/4” O/D x 16 SWG	DN35	40	50	scintiallator
8	3/4” O/D x 16 SWG	DN35	50	227	flood gun
9	4” O/D x 16 SWG	DN100	70	40	view port
10	3/4” O/D x 16	DN45	33	90	microscope camera
11	4” O/D x 2.5mm	DN100	90	90	motorised manipulator
12	3/4” O/D x 18 SWG	DN16	38	313	viewport for light source adaptor
13	3/4” O/D x 18 SWG	DN16	30	330	iris shutter drive (Field of View)[11]
14	3/4” O/D x 2.5mm	DN63	40	270	raster ion gun EX06
15	1/2” O/D x 18 SWG	DN35	34	204	11-pin feedthrough for flood gun
16	8” I/D x 14 SWG	DN200	-	-	turbomolecular pump
17	4” O/D x 16 SWG	DN100	90	221	magnetic lens assembly
18	1/2” O/D x 18 SWG	DN35	34	149	ultraviolet source

[11] iris shutter drive (Angle) is located upper

Appendix C: drawings of preparation chamber

(a) (b)

Figure 33: Preparation chamber: (a) top view, (b) side view

Table 4: Flanges and equipment of preparation chamber

No	Flange	Designation
1	DN63	analytical chamber
2	DN35	ion gun accessory
3	DN35	spare flange
4	DN35	ion gun EX03
5	DN35	heating state
6	DN35	vacuum-gauge sensor
7	DN35	differential pump-down
8	DN35	spare flange
9	DN63	viewport
10	DN35	gas line
11	DN63	spare flange
12	DN63	load lock chamber
13	DN16	viewport for light source adaptor
14	DN35	carrier of platform for placement of 3 sample holders
15	DN63	viewport
16	DN150	diffractometer Omicron SPECTALEED
17	DN63	VG Scienta 4-axis manipulator
18	DN63	viewport
19	DN35	manipulator
20	DN100	pump-down
21	DN35	spare flange