Laboratory X-ray reflectometer for liquid interfaces investigation

V.E. Asadchikov[1], V.V. Volkov[1], Yu.O. Volkov[1], K.A. Dembo[1], I.V. Kozhevnikov[1], B.S. Roshchin[1], A.M. Tikhonov[2], D.A. Frolov[1]
[1]A.V. Shubnikov Institute of Crystallography RAS, Moscow, Leninskii pr. 59
[2]P.L. Kapitza Institute for Physical Problems RAS, Moscow, Kosygina str. 2
e-mail: ross@crys.ras.ru

A.V. Shubnikov
Institute of Crystallography

Aleksey Vasil'yevich Shubnikov (1887 – 1970) was a founder of Russian crystallography.
He expanded crystallography from mineralogy to physics, chemistry and mathematics.
Institute of Crystallography developed from Shubnikov's crystallography laboratory which was founded for developing quartz single crystals growing technique.
Now Shubnikov Institute of crystallography consists of more than 20 departments which cover broad area of science: inorganic crystals growth, protein crystallography, X-ray diffractometry, reflectometry, scattering, scanning probe and electron microscopy, synchrotron radiation techniques, etc.

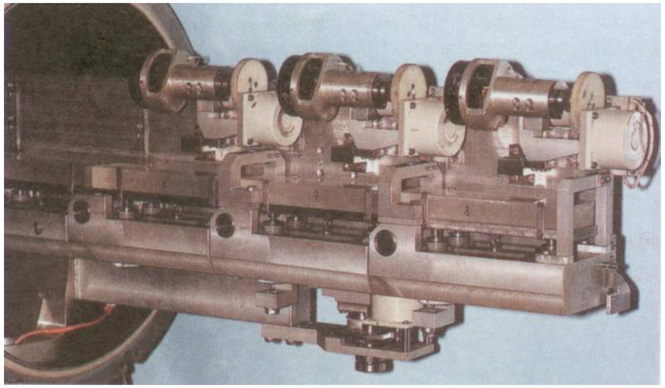
Design Department of SCI

Triple-crystal X-ray spectrometer

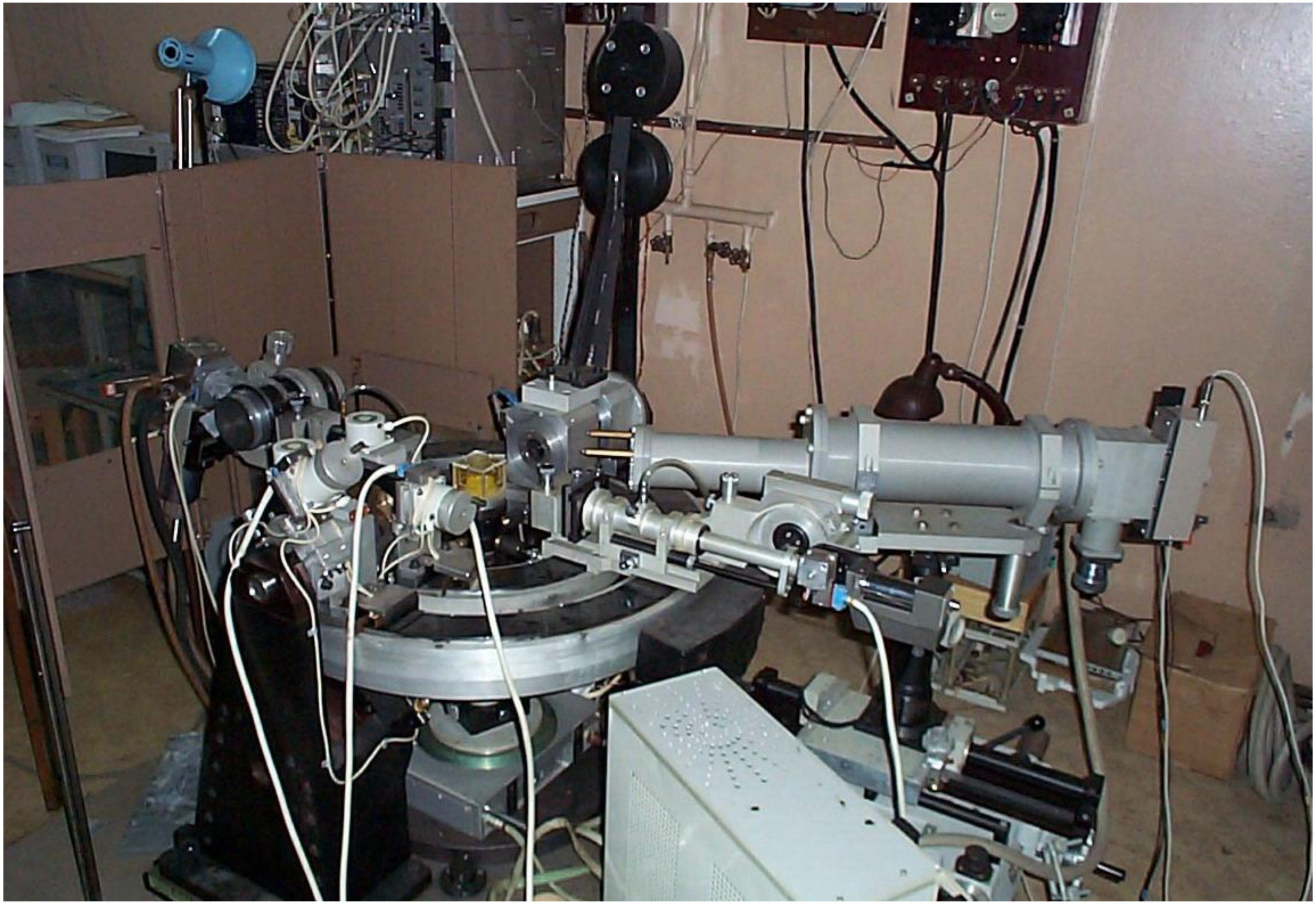
Crystallizer for
horizontal sapphire growth

Focusing monochromator
at EXAFS beamline

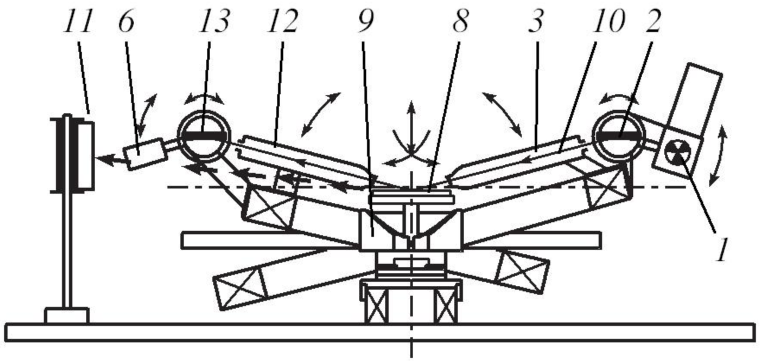
Focusing mirror of “Protein” beamline
at Kurchatov Institute Synchrotron Source

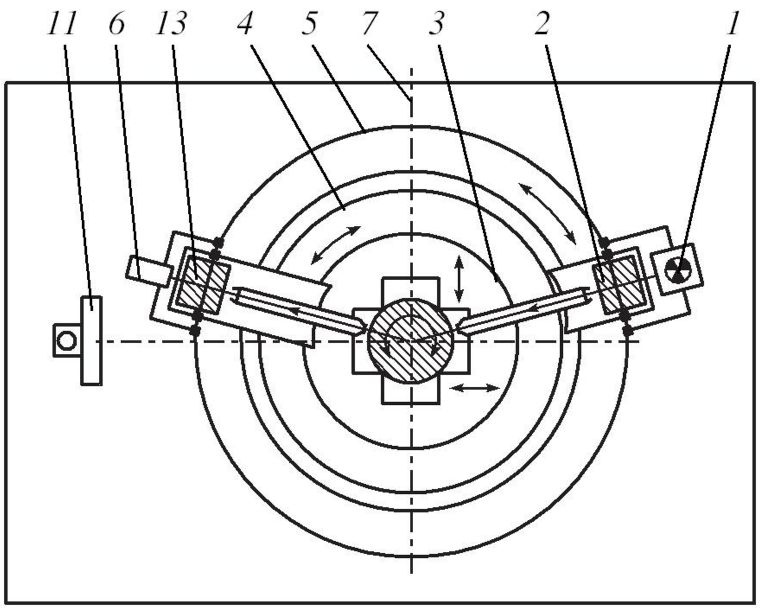


Overall view of the reflectometer



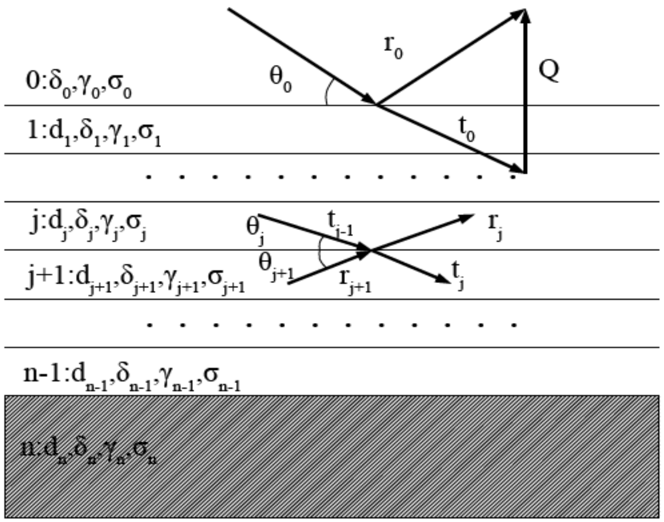
Reflectometer layout

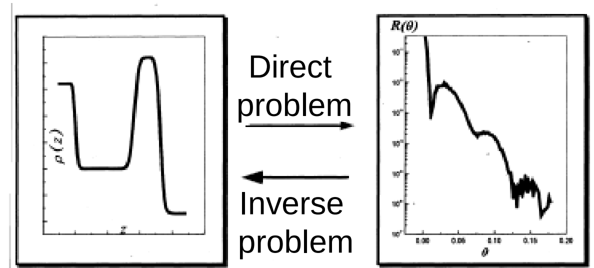


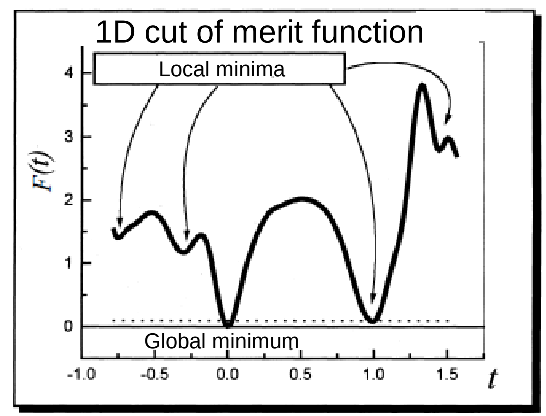


(1) X-ray tube;
(2) monochromator crystal;
(3, 12) collimating system;
(4) ring support for the X-ray tube;
(5) ring support for the detector;
(6) scintillation detector;
(7) rotation axis of ring supports 4 and 5;
(8) test sample;
(9) sample holder with the alignment table;
(10) X-ray beam;
(11) position-sensitive linear detector;
(13) analyzer crystal.

Inverse problem of X-ray reflectometry





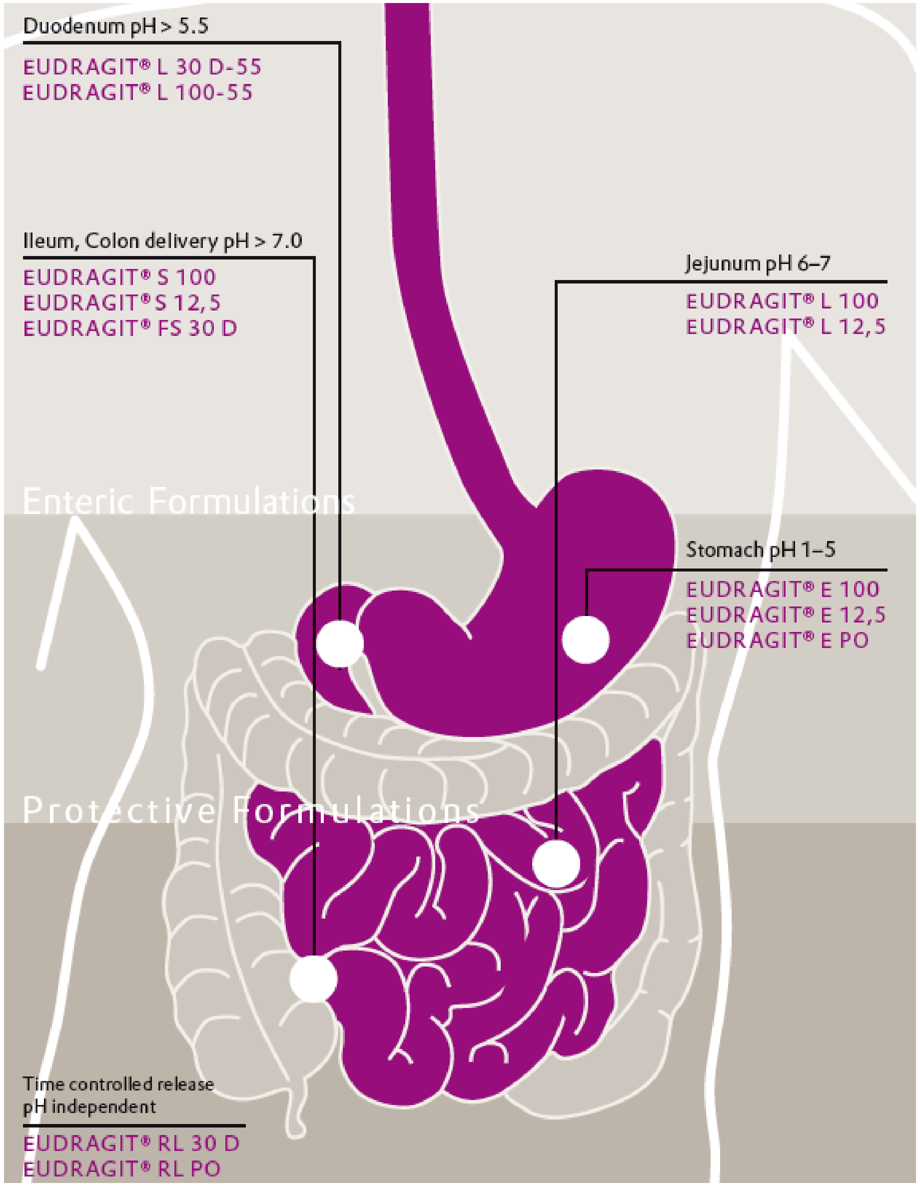


$$r_j = \frac{r_j^F + r_{j+1}e^{2i\chi_{j+1}d_{j+1}}}{1 + r_j^F r_{j+1}e^{2i\chi_{j+1}d_{j+1}}}; \quad r_{n+1} = 0; \quad j = 0, 1, \cdots, n;$$

$$MF(\delta_1, \ldots, \delta_{N+1}, N) = \sum_{j=1}^{M} \left[\log R(\theta_j) - \log R_{exp}(\theta_j)\right]^2 + Q \sum_{\substack{i=2,\ldots,N-1 \\ i \neq i_1, i_2, \ldots, i_m}} (\delta_{i+1} - \delta_i)^2,$$

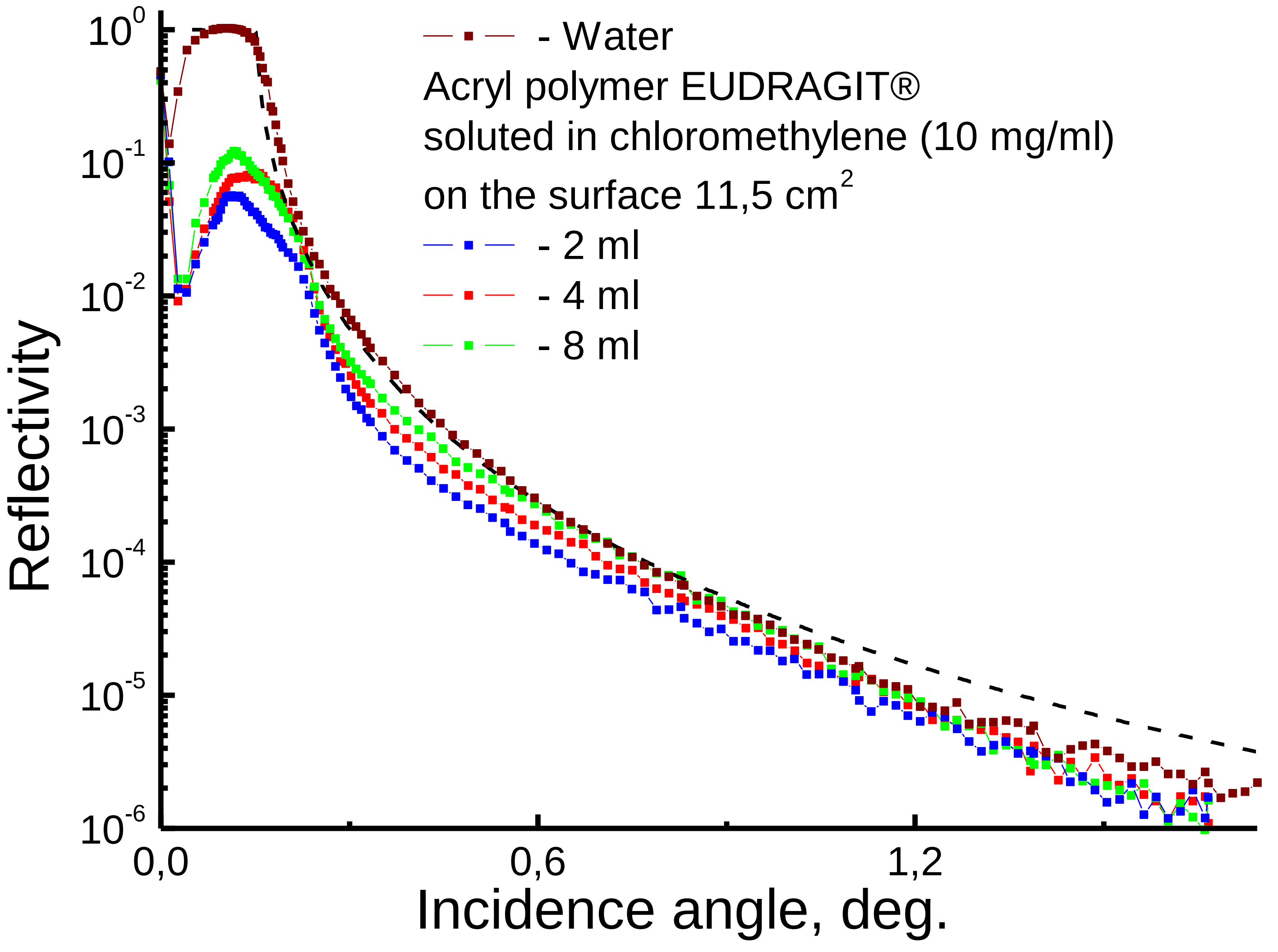
I.V. Kozhevnikov. Physical analysis of the inverse problem of X-ray reflectometry // Nuclear Instruments and Methods in Physics Research A.— 2003.— Vol. 508.— Pp. 519–541.

Medical applications of acryl polymers



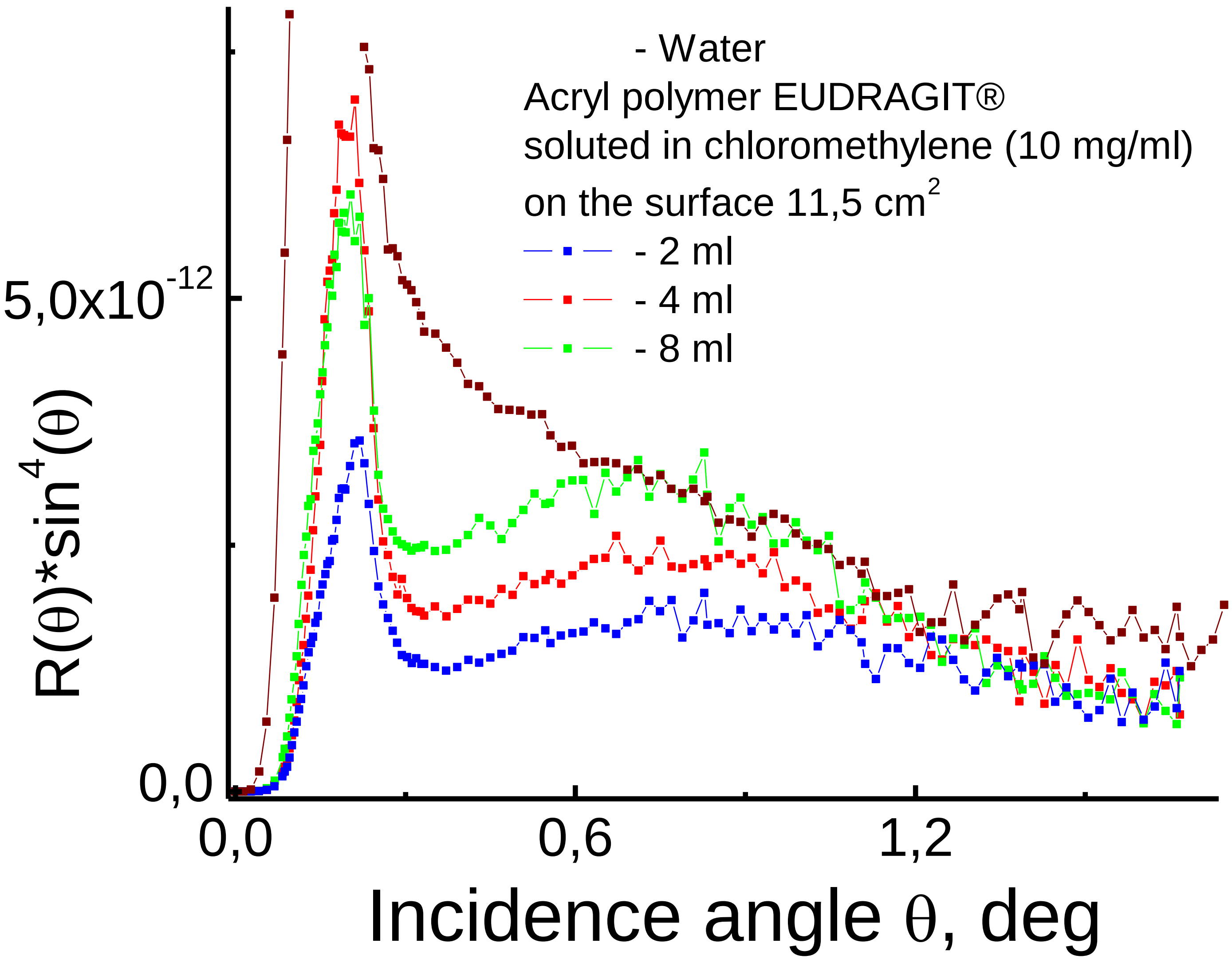
http://eudragit.evonik.com

Reflectometry of polymer layers on water



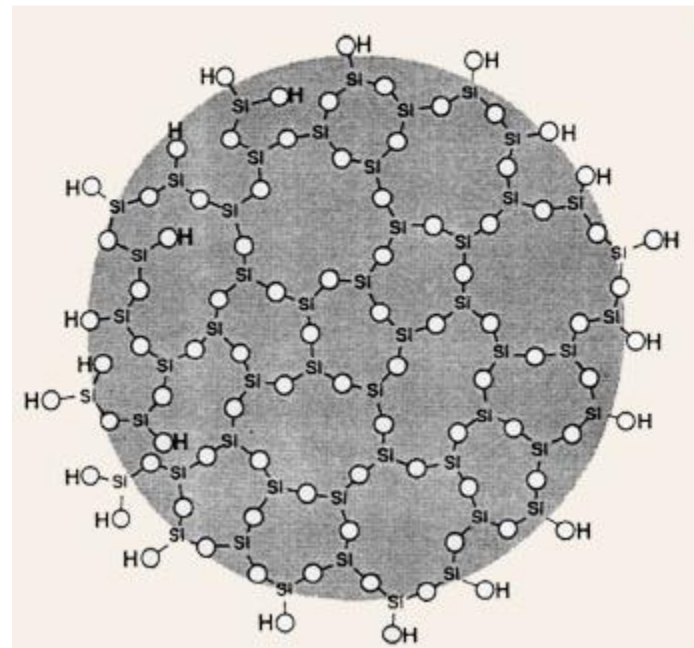
V.E. Asadchikov, V.G. Babak, A.V. Buzmakov et al // Instruments and Experimental Techniques (in Russian), 2005, V. 3, PP. 99–107

Reflectometry of polymer layers on water

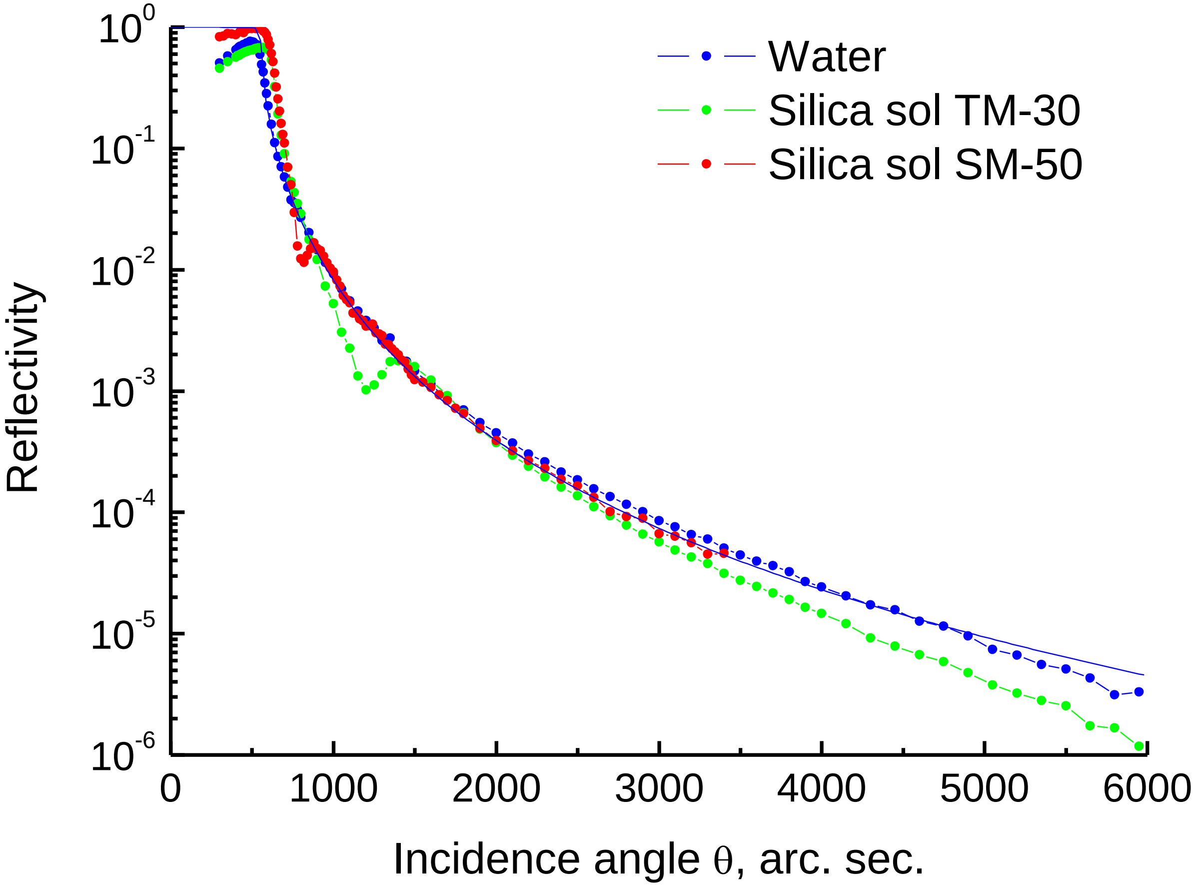


V.E. Asadchikov, V.G. Babak, A.V. Buzmakov et al // Instruments and Experimental Techniques (in Russian), 2005, V. 3, PP. 99–107

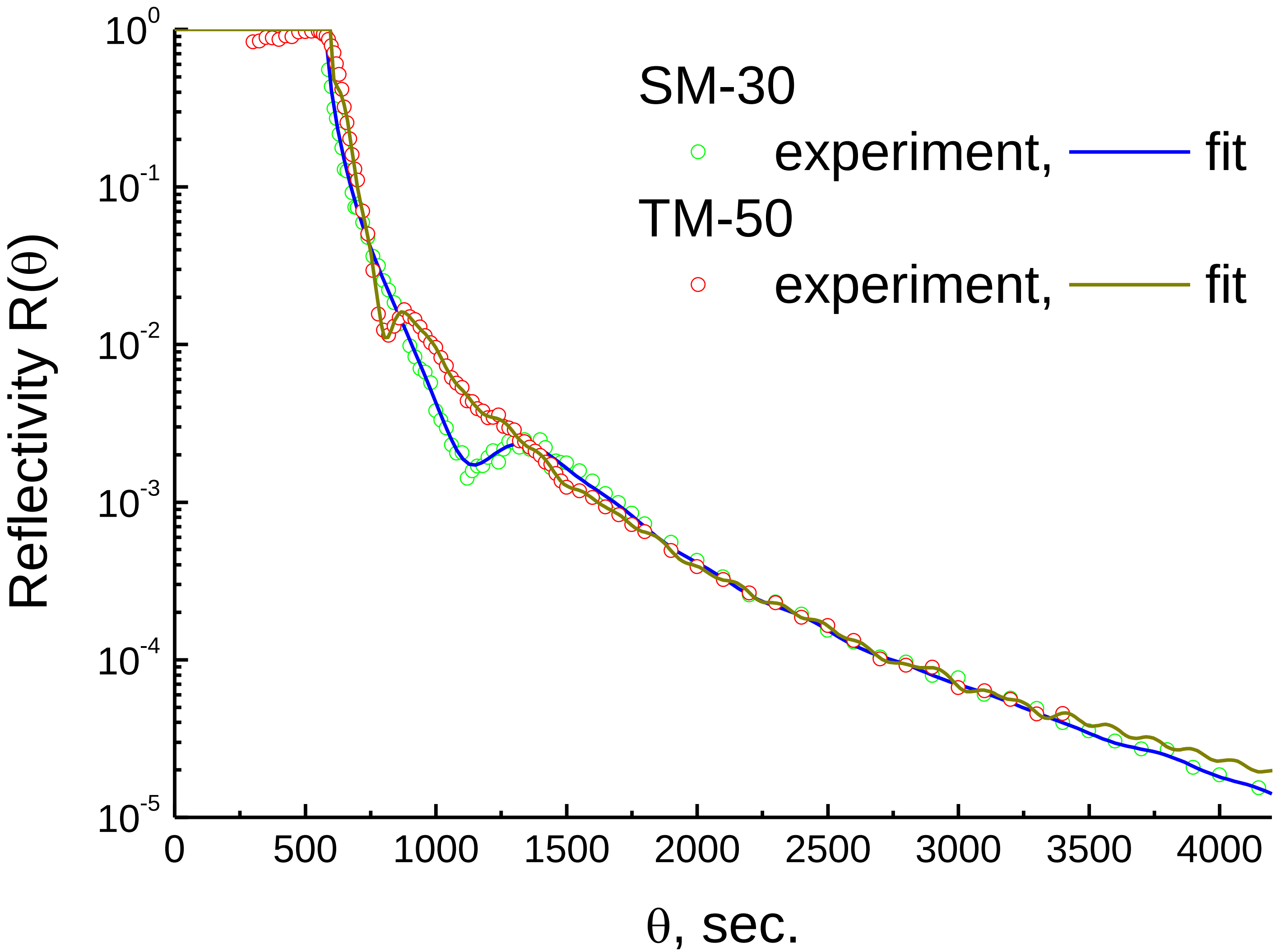
Silica sol – colloidal solution of SiO_2 nanoparticles in water



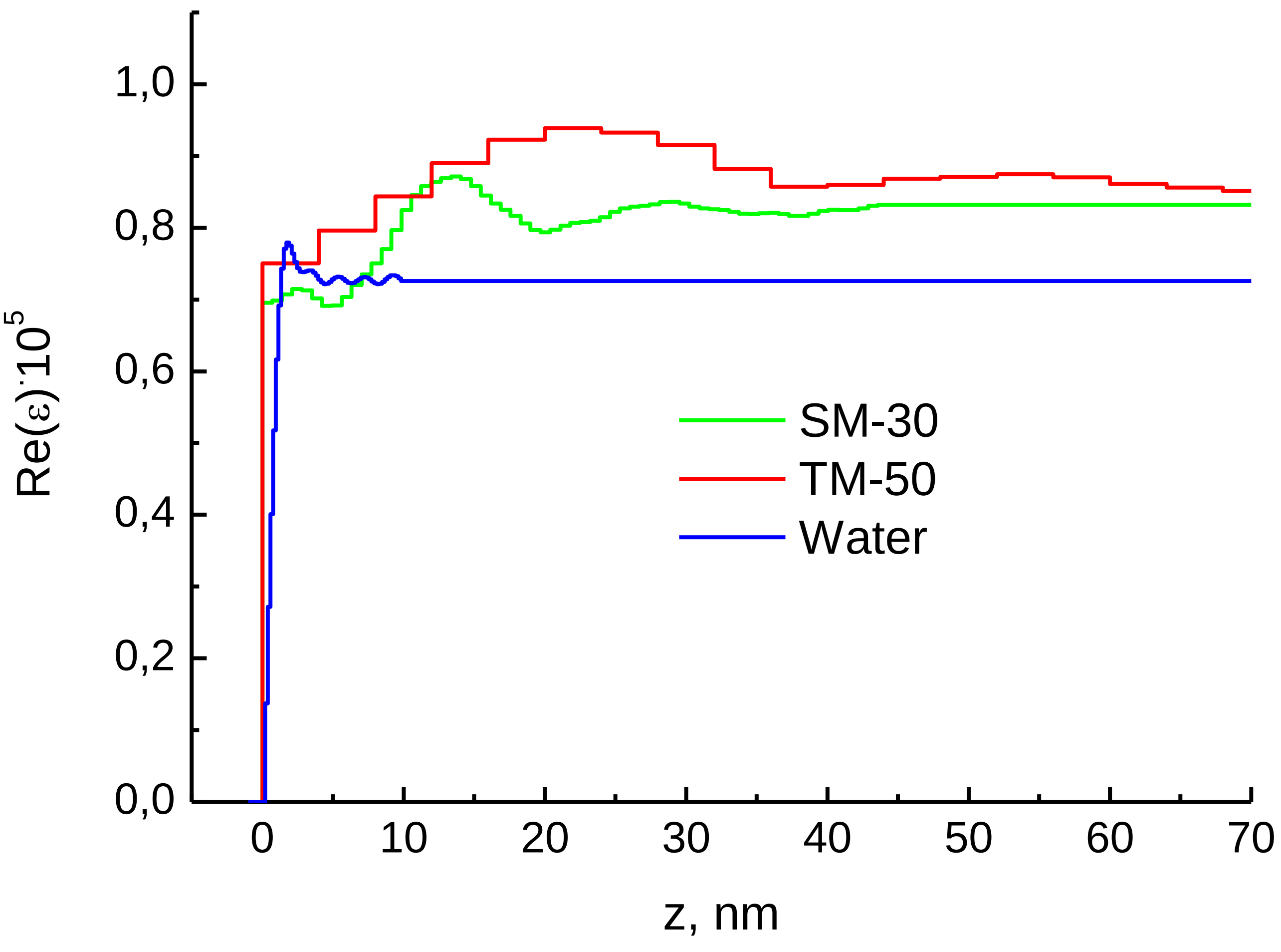
Reflectometry of water and silica sol surfaces



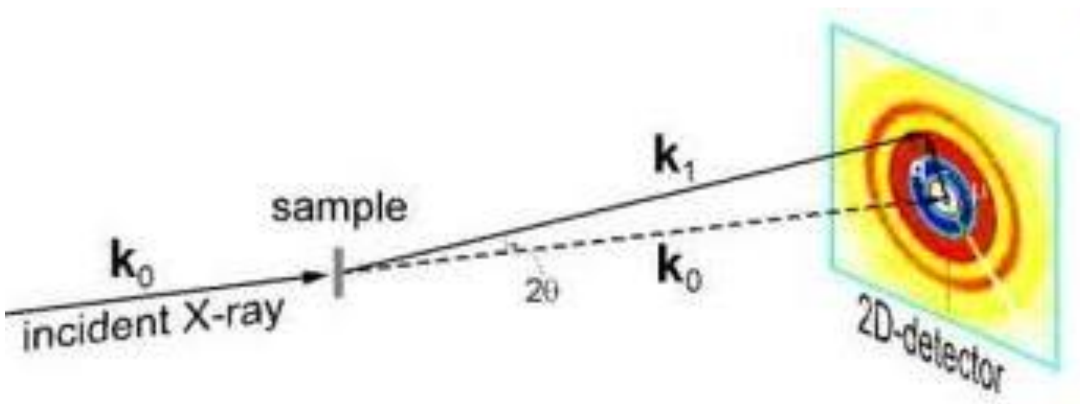
Dielectric permeability profile reconstruction for silica sol

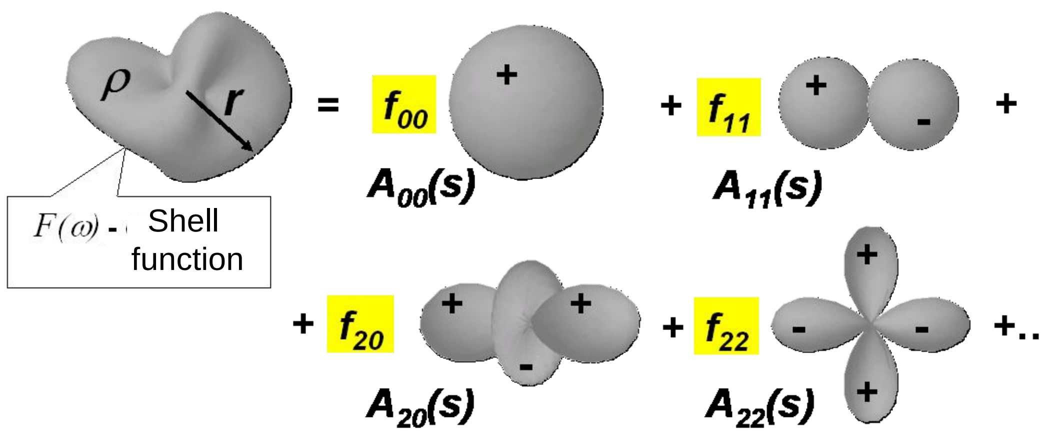


Dielectric permeability profile reconstruction for silica sol

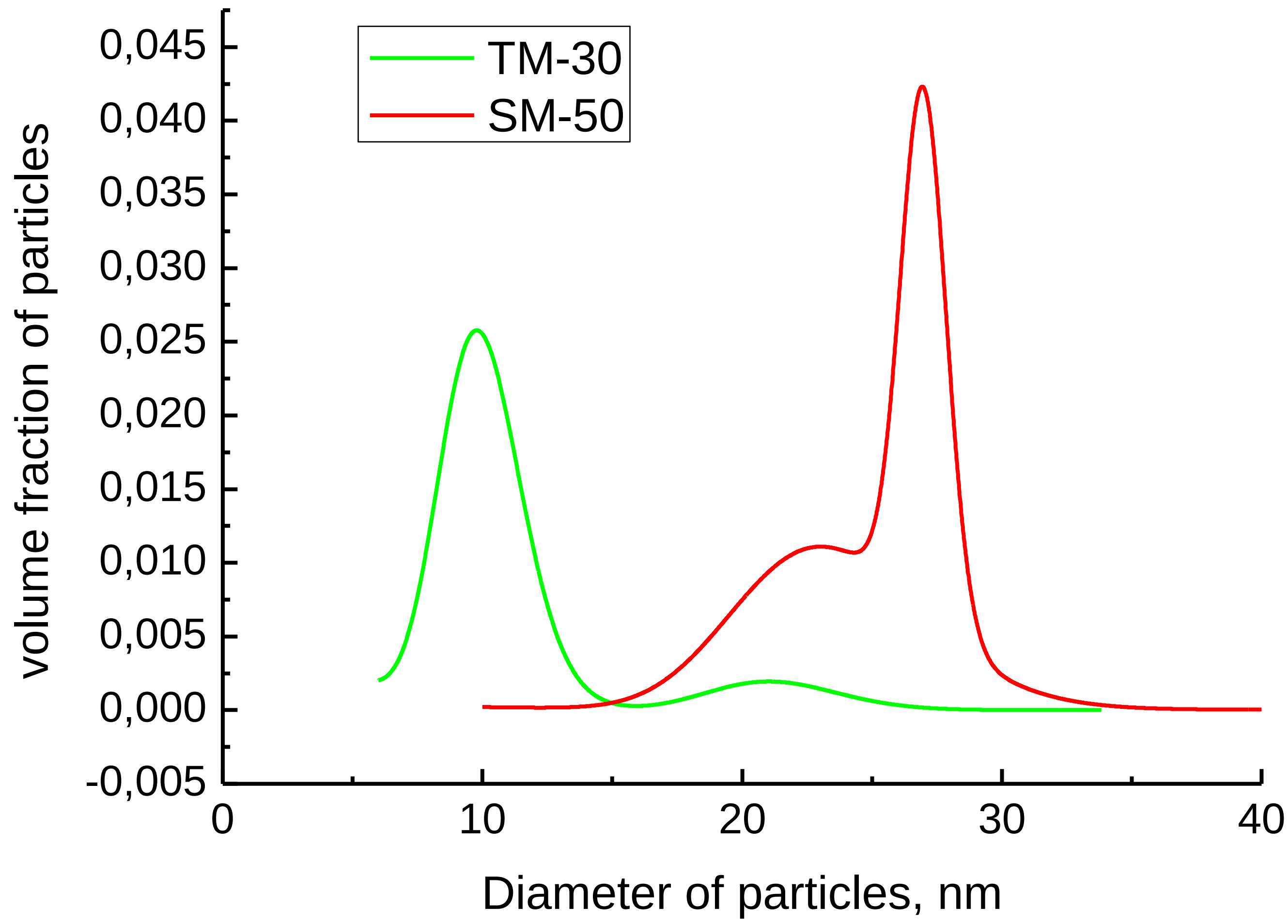


Small-angle scattering

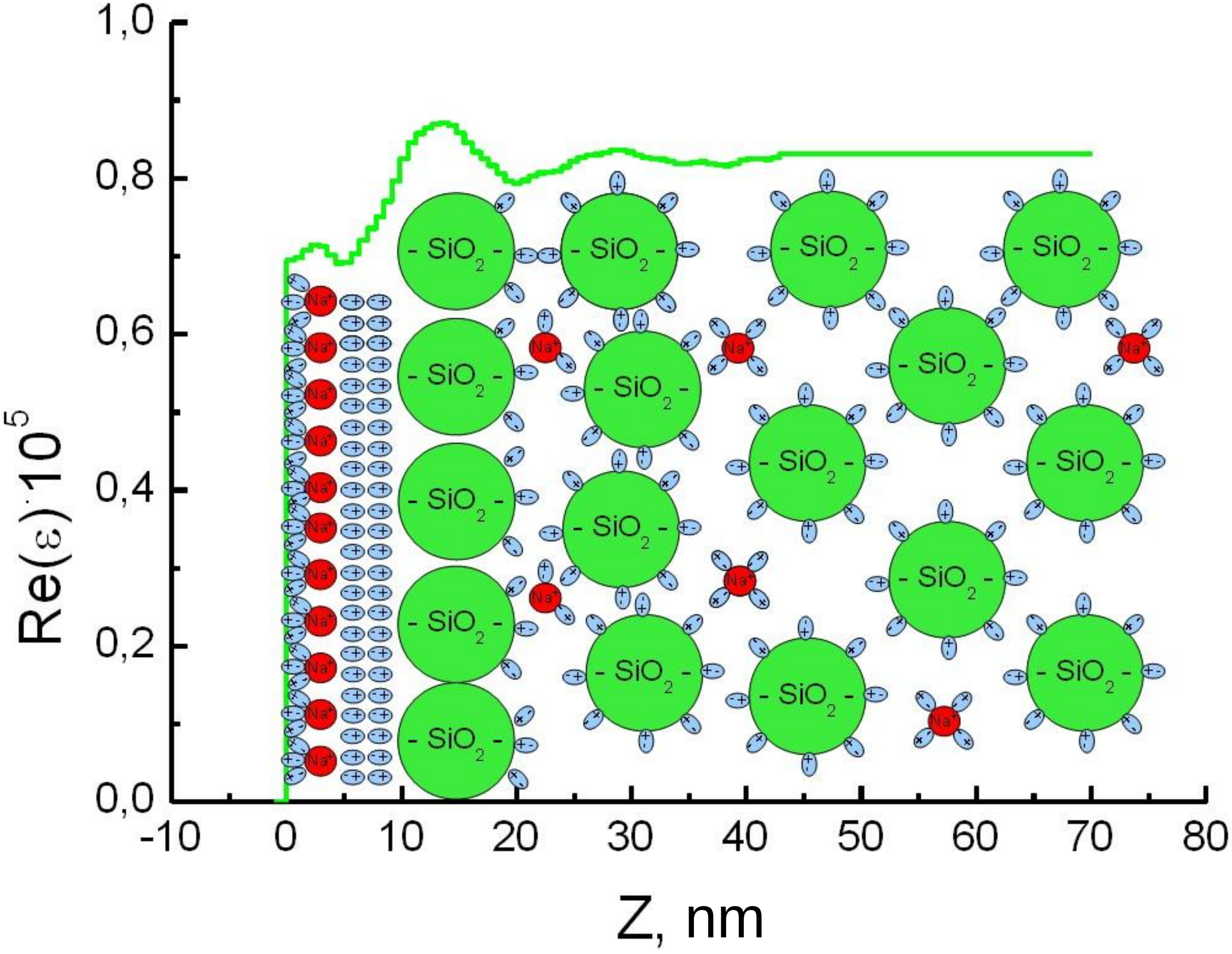




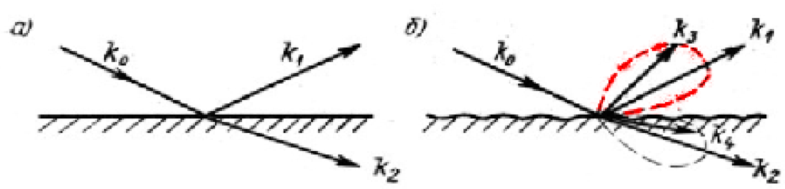
Size-dispersion of SiO_2 particles in sol obtained by SAXS

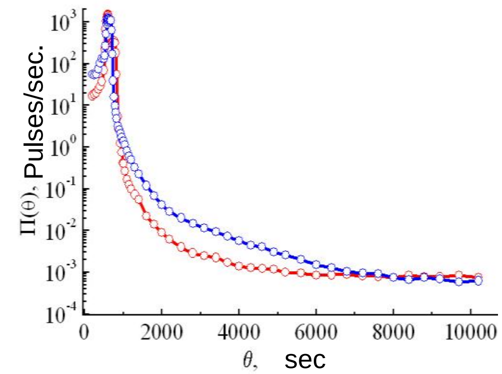


Double ionic layer formation

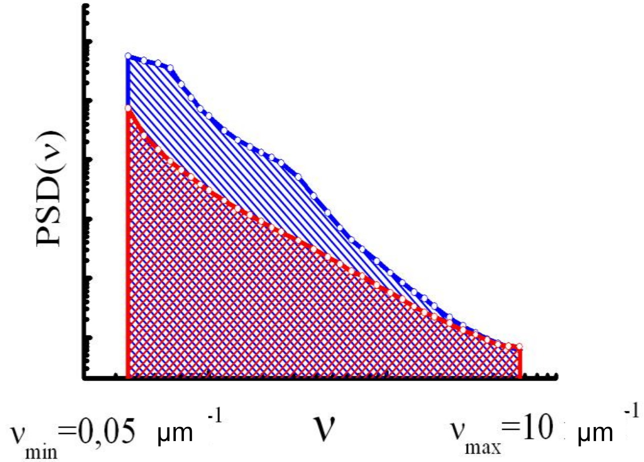


Grazing incidence small-angle scattering



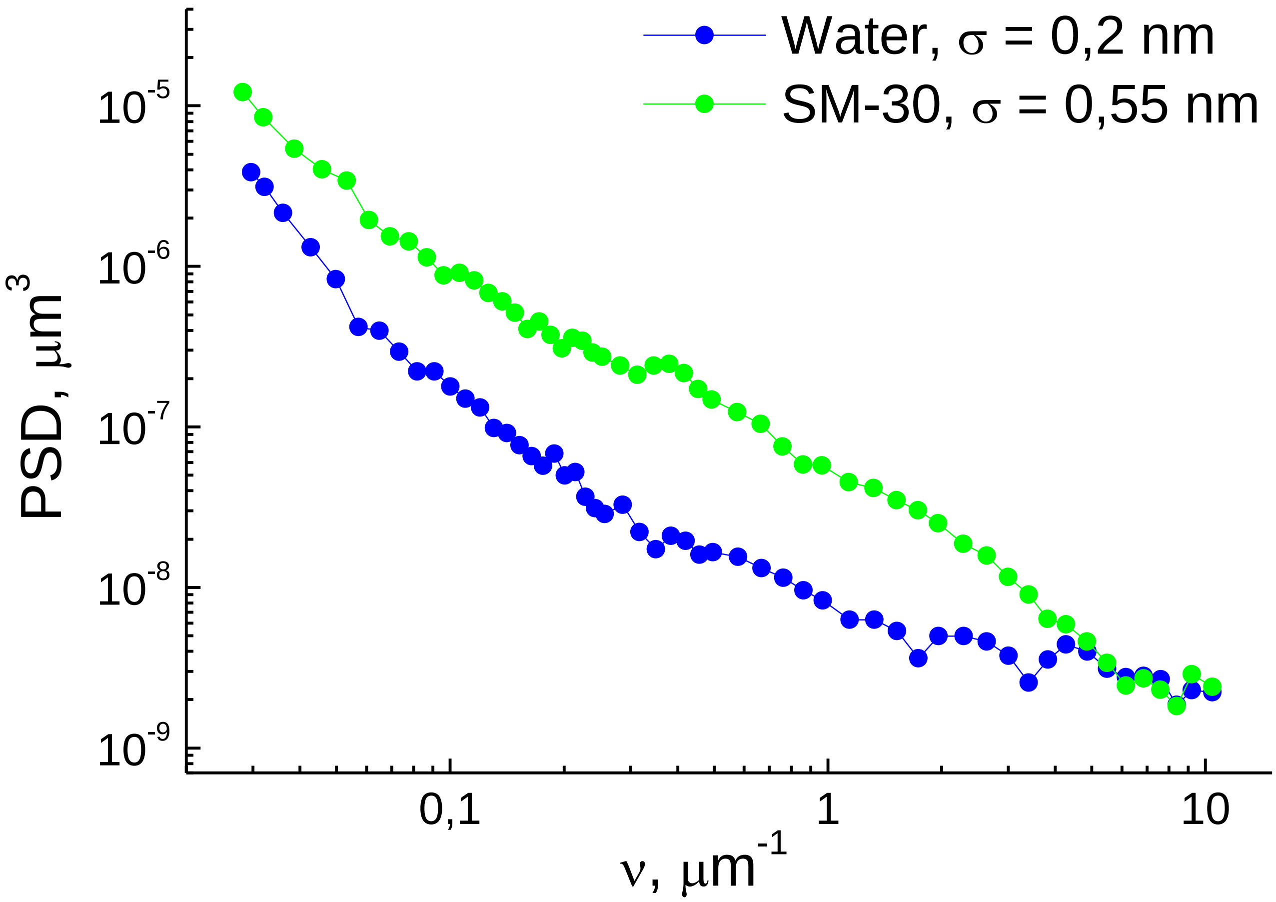


$$\Pi(\theta) = \int \Phi(\theta,\phi)d\phi = \frac{1}{W_{inc}}\frac{dW_{scat}}{d\theta} = \frac{k^3|1-\varepsilon_+|^2|t(\theta_0)t(\theta)|^2}{16\pi\sin\theta_0\sqrt{\cos\theta_0\cos\theta}} \times PSD_{1D}(\nu); \quad (1.12)$$

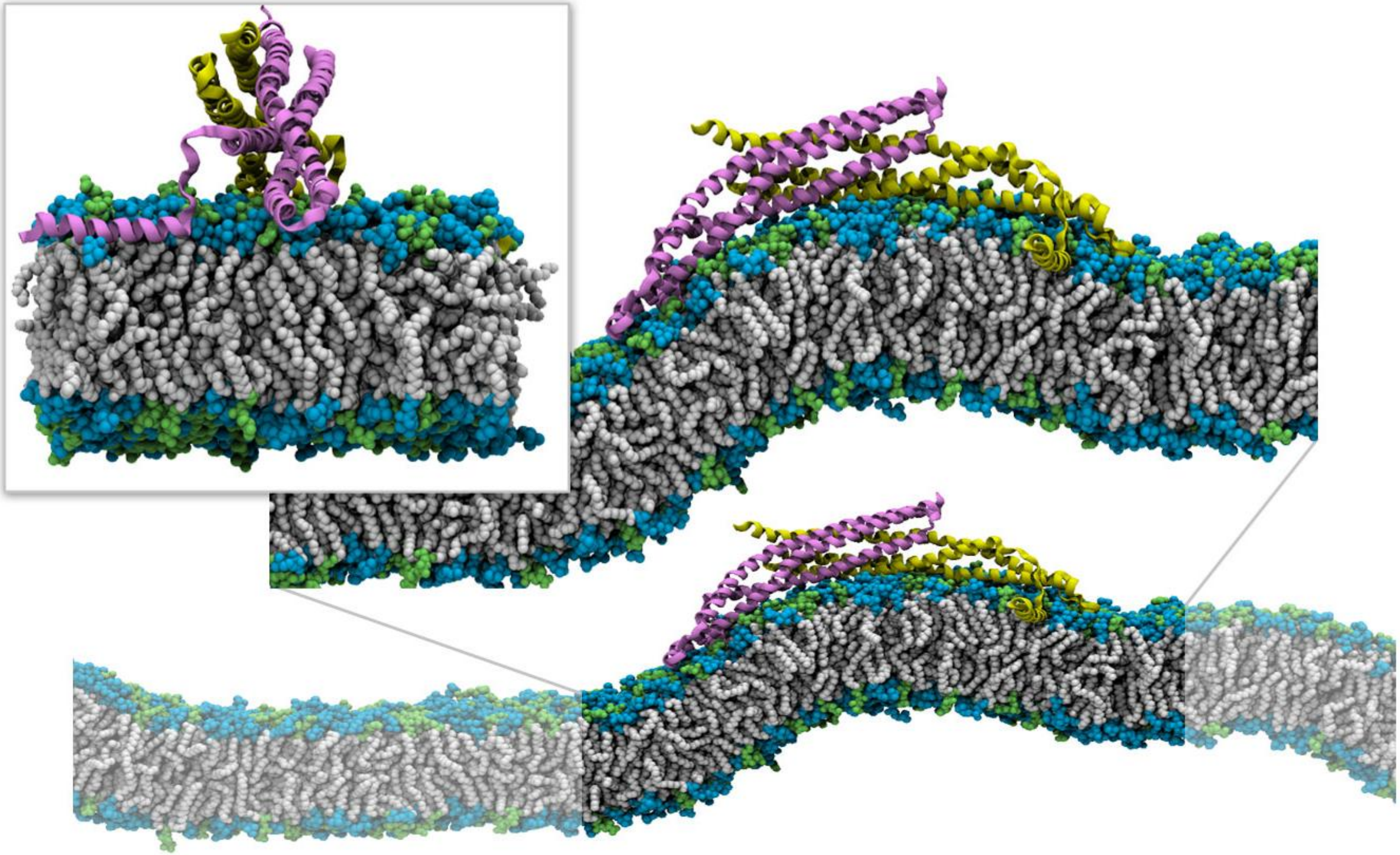


$$\sigma^2_{eff} = \int_{\nu_{min}}^{\nu_{max}} PSD(\nu)d\nu$$

Scattering on the silica sol surface

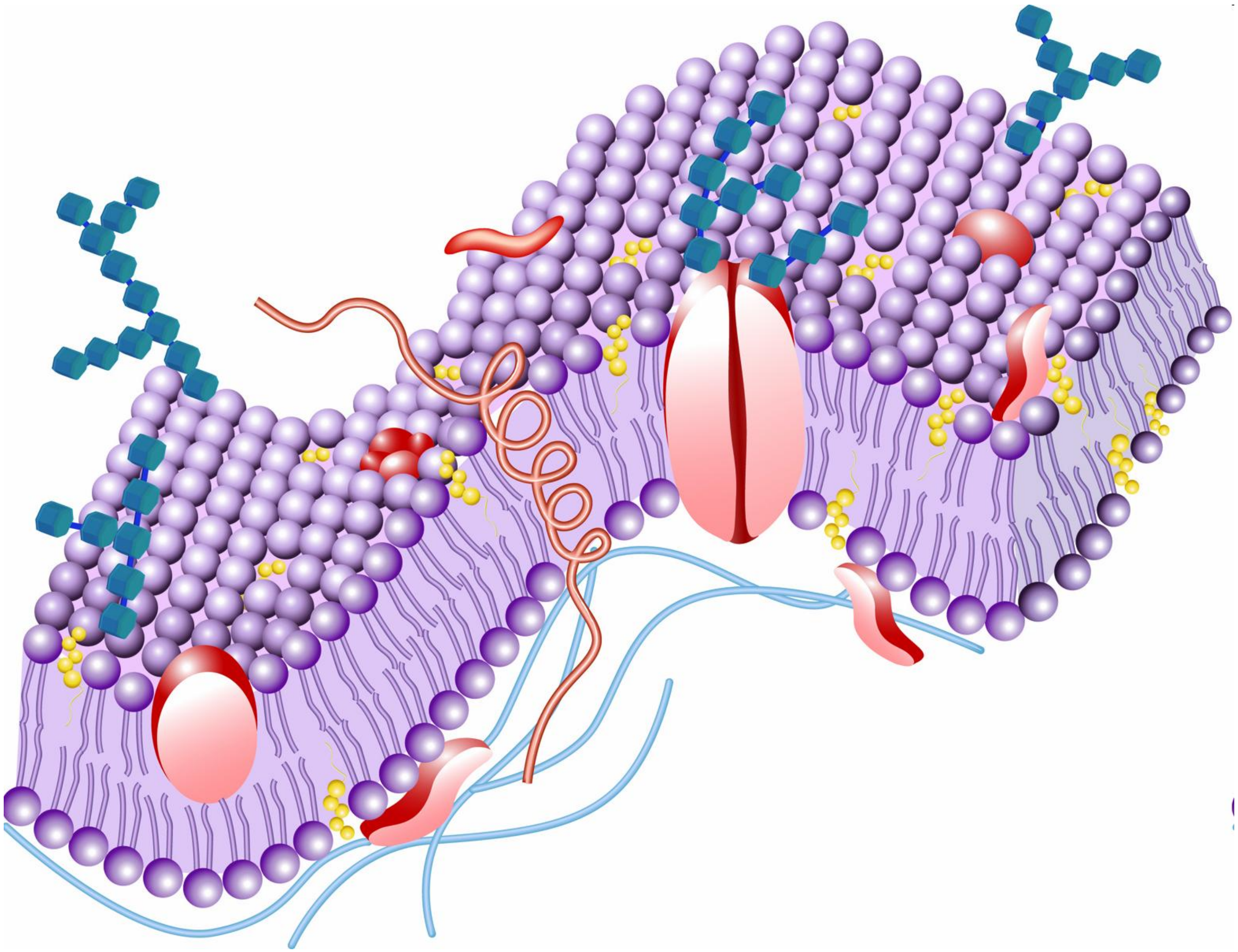


Lipid membranes



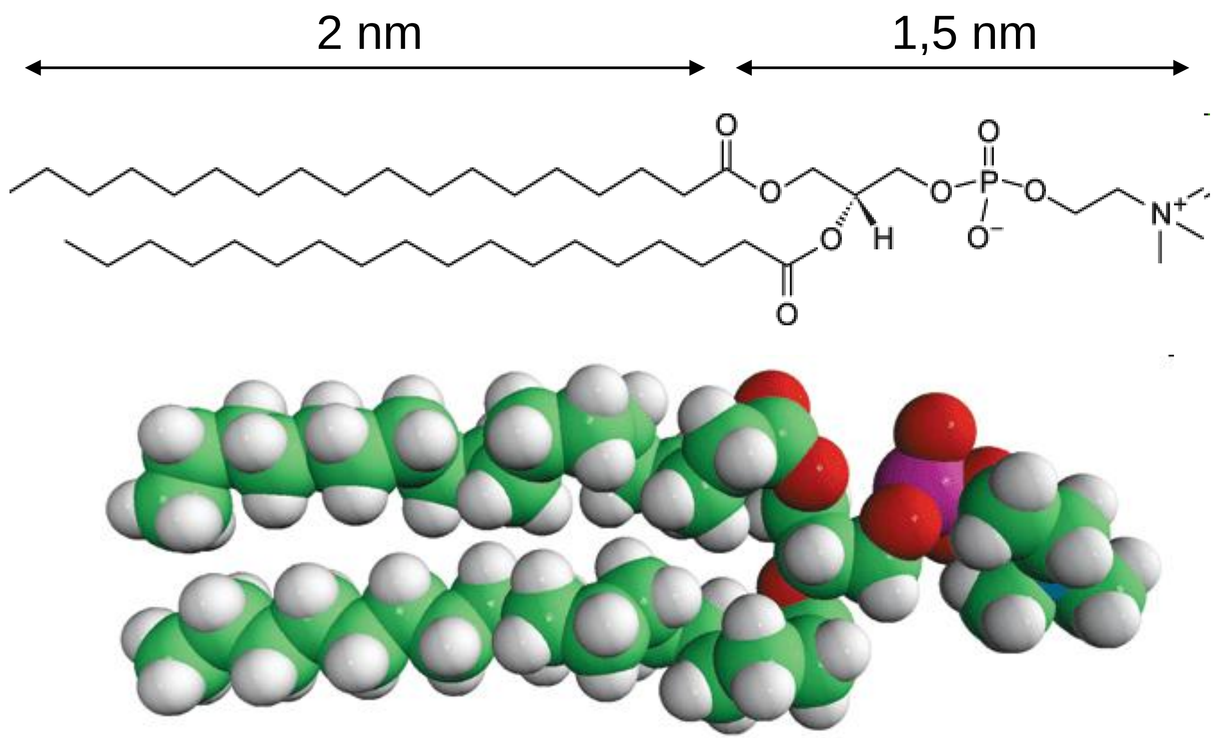
http://www.psc.edu/science/2007/bardomain/

Lipid membranes



http://arisumi-illustration.com/blog

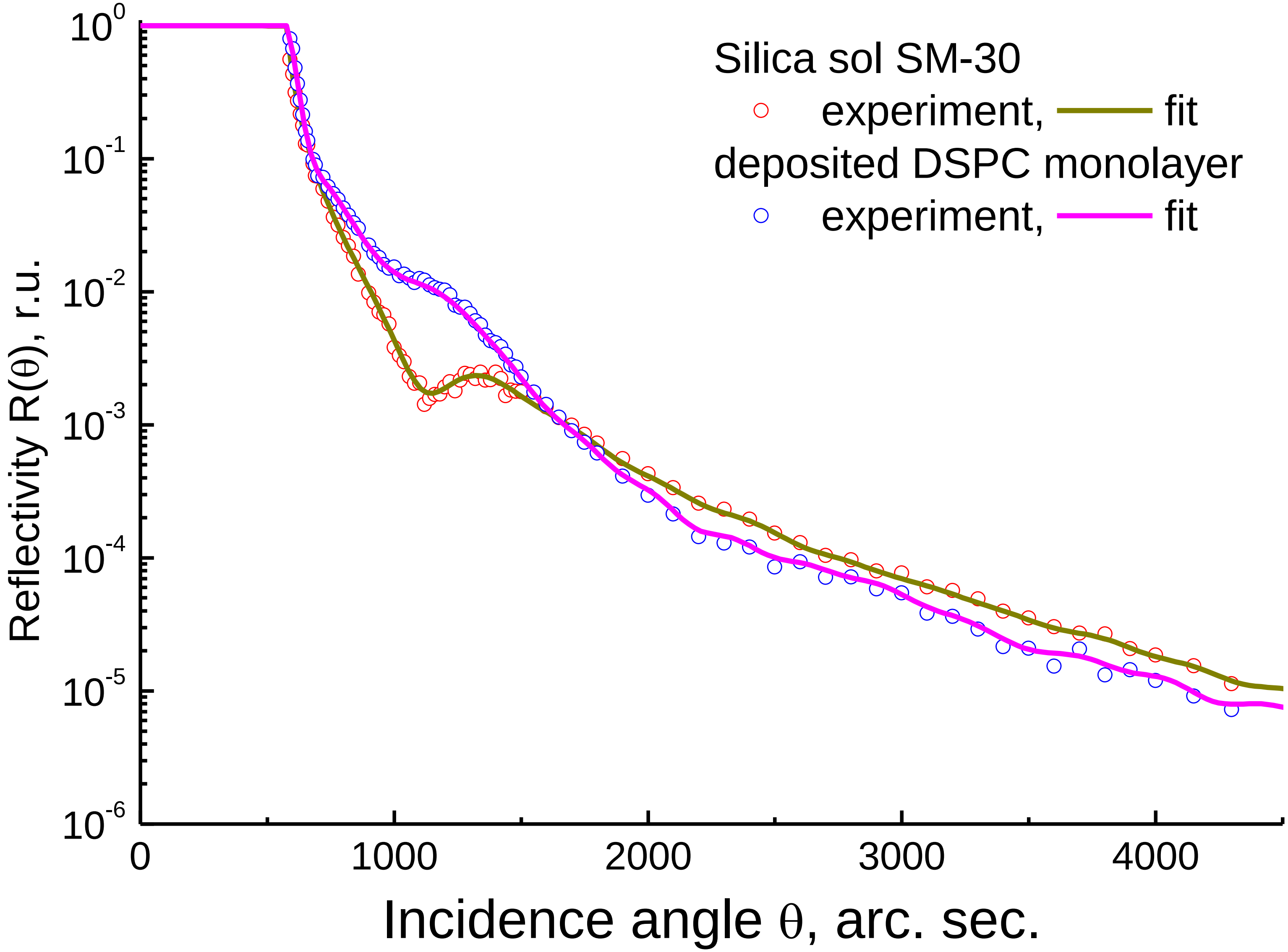
1,2-Distearoyl-sn-glycero-3-phosphocholine (DSPC)



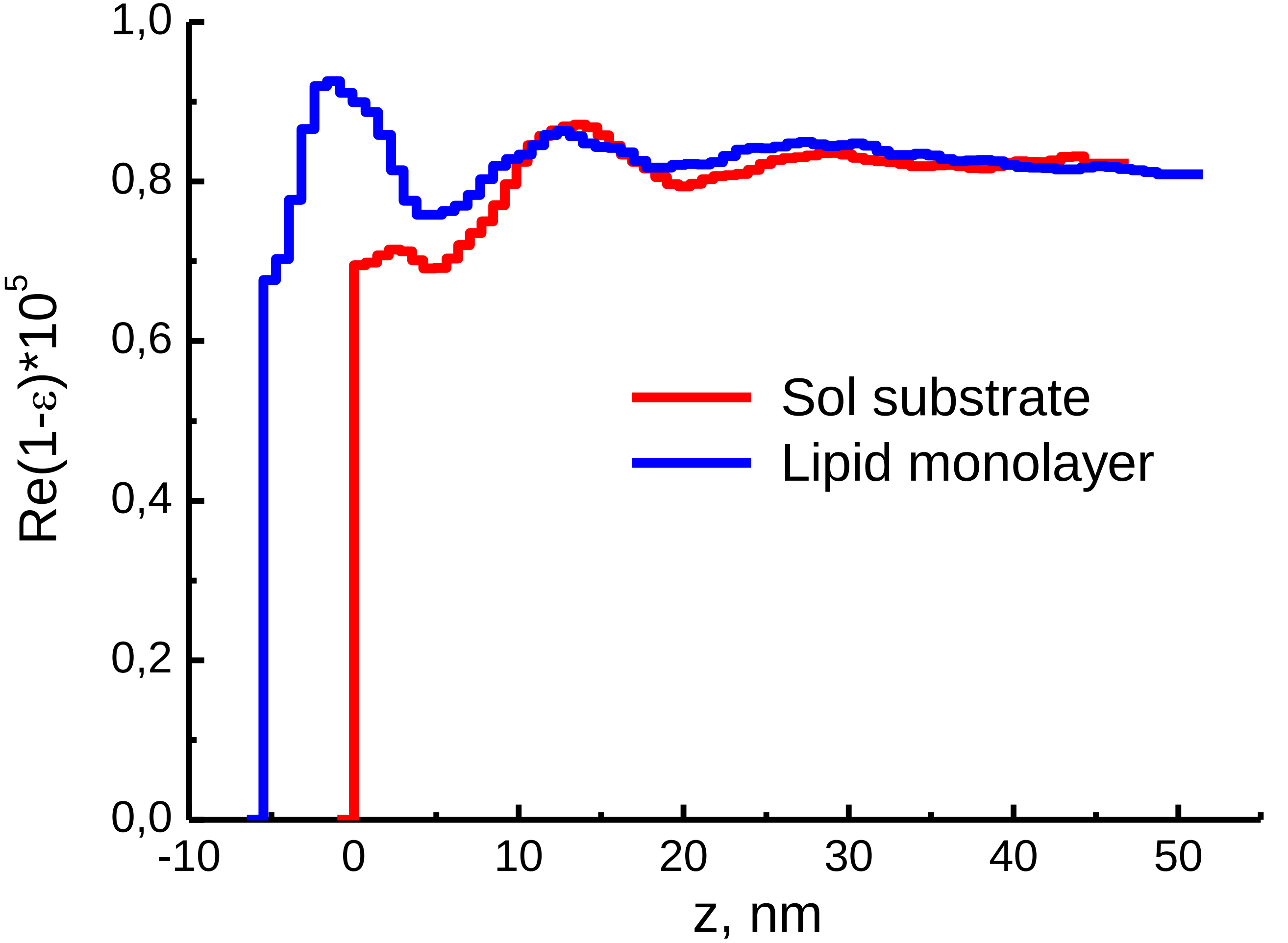
Formula	$C_{44}H_{88}NO_8P$
Molecular weight	790.145

http://www.avantilipids.com

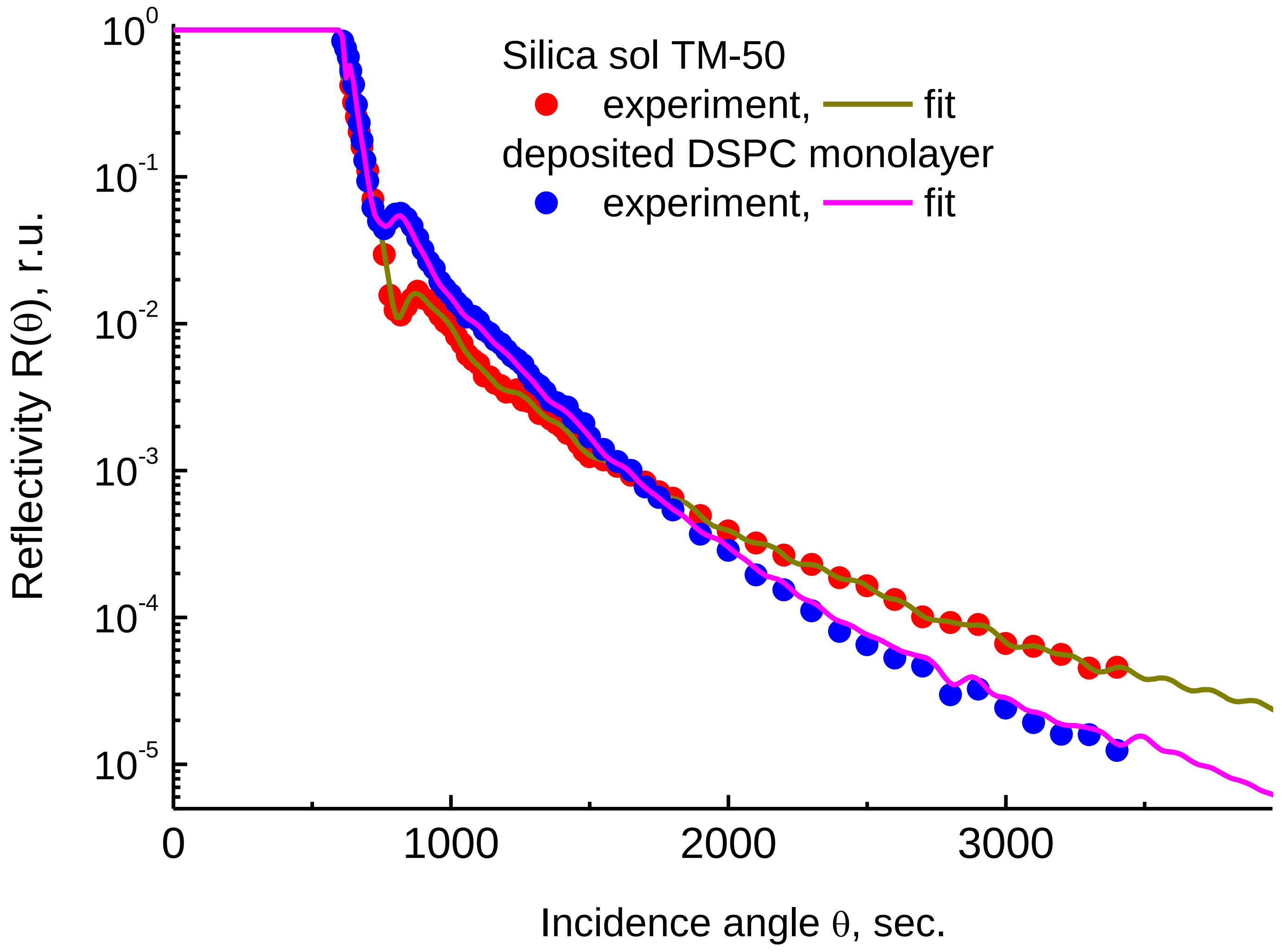
DSPC monolayer on SM-30 sol



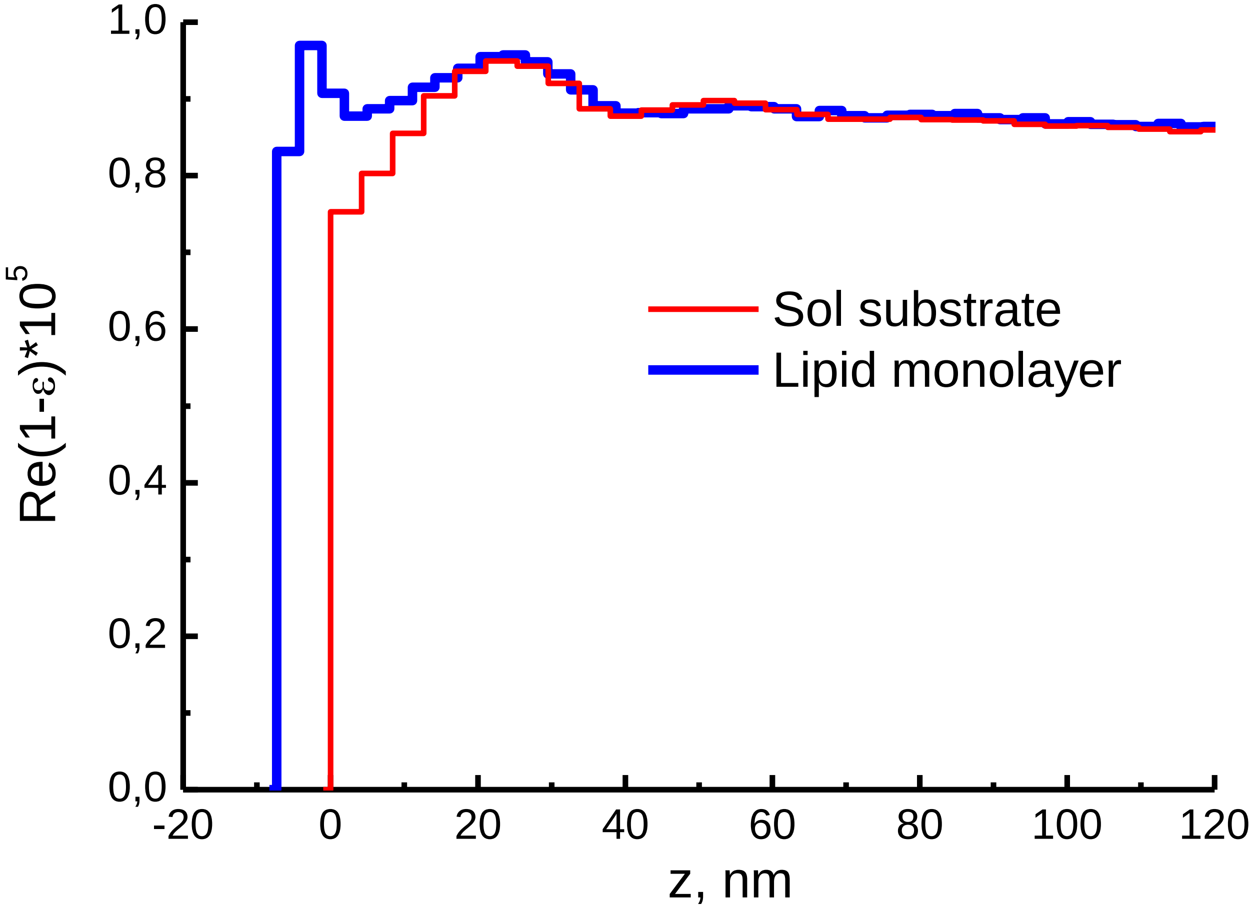
DSPC monolayer on SM-30 sol

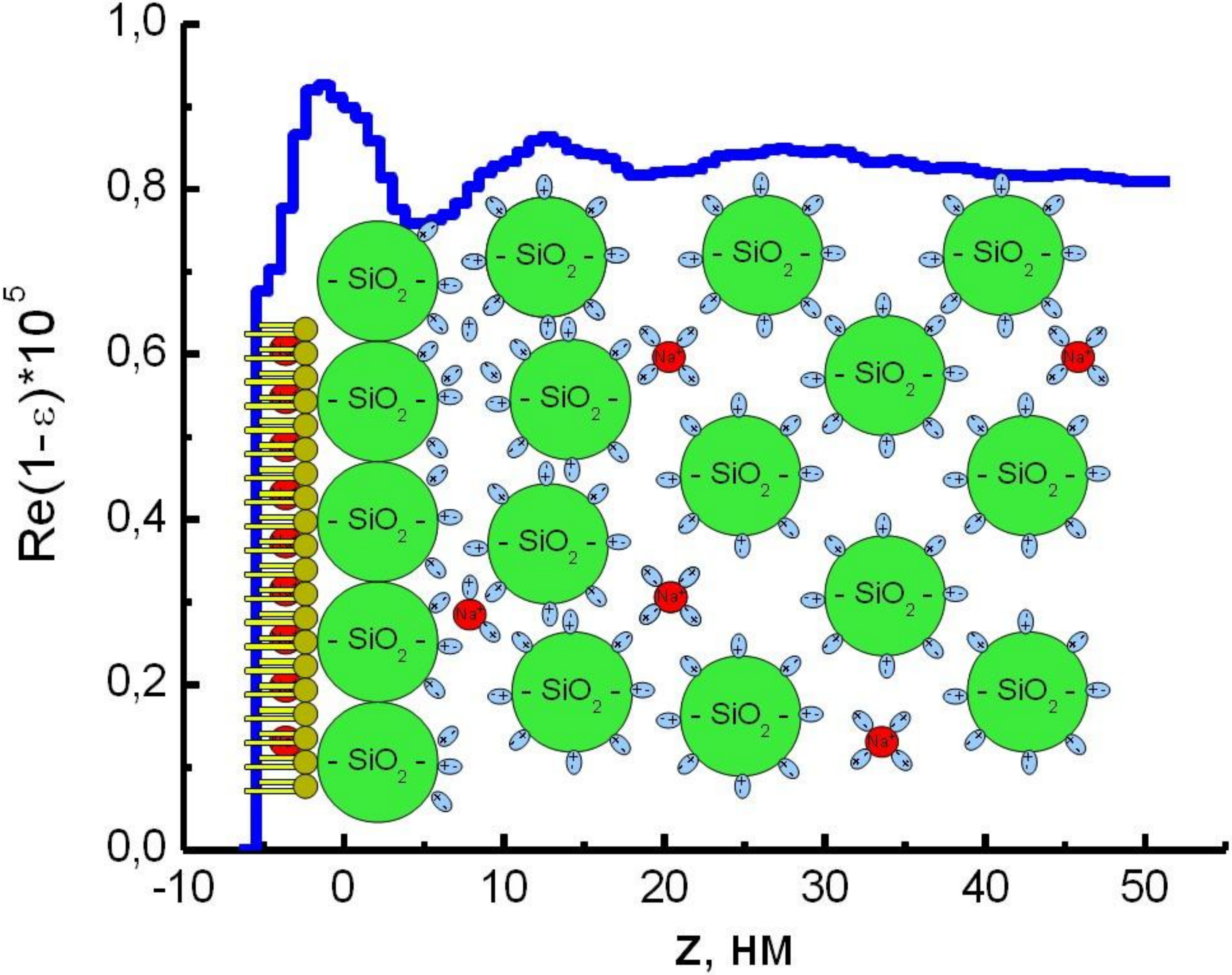


DSPC monolayer on TM-50 sol



DSPC monolayer on TM-50 sol




1,0
0,8
0,6
0,4
0,2
0,0
Re(1-ε)*10^5
-10
0
10
20
30
40
50
Z, HM
SiO2
Na+

Conclusion

- Laboratory reflectometer provided complex X-ray technique applied. It is revealed that silica sol has near-surface structure of several layers which depth is more than 50% higher than it was previously thought.
- The possibility to use silica sol as a substrate for lipid monolayers is shown.
- The raising of near-surface SiO_2 particles density while deposing lipid on the sol surface is found. It is determined apparently by positive potential formation caused by Na^+ ion diffusion into the lipid monolayer.

Thank you for attention!