Tetrahedron Letters No. 6, pp 593 - 594, 1978. Pergamon Press. Printed in Great Britain.

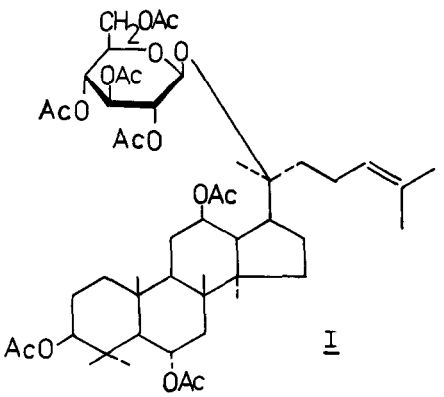
X-RAY ANALYSIS OF PANAXOSIDE A PROGENIN I ACETATE, A TRITERPENE GLYCOSIDE ISOLATED FROM PANAX GINSENG C.A.MEYER

S.G.Iljin*, A.K.Dzizenko and G.B.Elyakov
Pacific Institute of Bioorganic Chemistry, Far East Science Center,
USSR Academy of Sciences, Vladivostok 22, U.S.S.R.

B.L.Tarnopolsky and Z.S.Safina
Institute of Chemical Physics, Academy of Sciences, Moscow, U.S.S.R.

(Received in UK 12 December 1977; accepted for publication 16 December 1977)

The structures of ginseng glycosides have been widely discussed in the last fifteen years[1-6]. An X-ray analysis of panaxoside A progenin I acetate (I) has been carried out for the first time. In this article we report the crystal data and conformation of I (more detailed results will be published later).



I

Crystal data. $C_{50}H_{76}O_{16}$, M=933.15. Monoclinic, a=8.698(5), b=11.736(5), c=26.700(10) Å, γ=92.884(17)°, V=2725 Å^3, Z=2, D_c=1.137 g/cm^3, F(000)=1008, Cu-K_α radiation, λ=1.5418 Å. Space group $P2_1$ from the systematic absences: 001, when $l \neq 2n$.

Crystallographic measurements. A crystal 0.08x 0.20x0.33 mm was grown from solution in ethyl acetate and used for all subsequent X-ray measurements. Preliminary cell dimensions and space group information were obtained from oscillation and precession photographs. The crystal was then transferred to a DRON-1 diffractometer, where accurate cell parameters were derived. Intensities with $\theta \leq 62°$ were obtained by the θ-2θ scanning technique on a DARM-2 automated diffractometer (Ni-filtered Cu-K_α radiation, equi-inclination method). From 2157 measurements, 1908 reflections with $I \geq 3\sigma$ were used in the analysis. The data were corrected for Lorentz-polarization effects, placed on an absolute scale by Wilson's method[7] and converted to normalized structure factors.

Structure analysis. The structure was solved by direct methods with Roentgen-75 programs[8] based on the multisolution tangent procedure[9]. The determination of the structure in an automatic regime was not a success, but a somewhat altered initial set of reflections yielded a reasonable E-map in which 40 of the 66 non-hydrogen atoms could be located. R corresponding to this synthesis was the lowest, 0.35. Successive Fourier syntheses defined the remaining C and O atoms.

Least-squares refinement of atomic coordinates, isotropic thermal para-

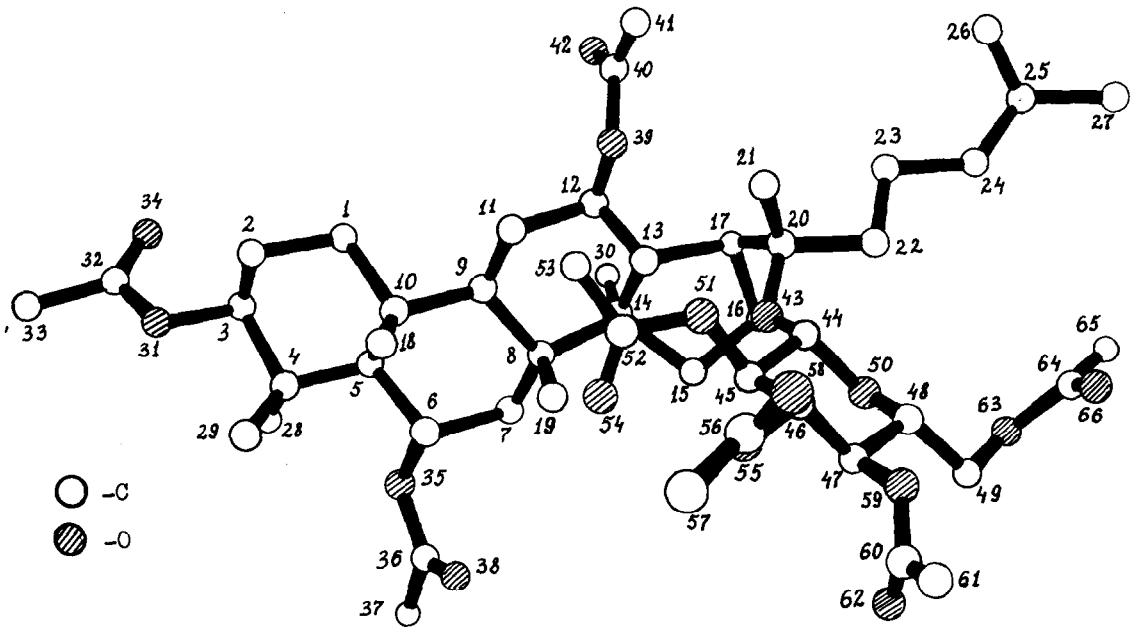


Figure 1. Conformation of I and numbering of the atoms.

meters and the scale factor gave R=0.16. Full-matrix refinement, including anisotropic thermal parameters, reduced R to 0.12.

The absolute configuration of I was defined in accordance with the conformation of D-glucose. The conformation is shown in Fig.1.

Acknowledges. Thanks are due to V.E.Zavodnik and V.I.Andrianov for their contribution to the X-ray analysis.

REFERENCES

1. G.B.Elyakov, L.I.Strigina, A.Y.Khorlin, N,K.Kochetkov, Izvest.Acad.Nauk SSSR, Otd.khim.n., 2054 (1962).
2. G.B.Elyakov, L.I.Strigina, N.K.Kochetkov, Dokl.Acad.Nauk SSSR, 158,892(1964).
3. M.Nagai, T.Ando, O.Tanaka, S.Shibata, Tetrahedron Lett., 3579 (1967).
4. Y.Iida, O.Tanaka, S.Shibata, Tetrahedron Lett., 5449 (1968).
5. Y.Nagai, O.Tanaka, S.Shibata, Tetrahedron, 27, 881 (1971).
6. L.I.Strigina, T.M.Remennikova, Y.N.Elkin, A.K.Dzizenko, V.V.Isakov, G.B.Elyakov, Dokl.Acad.Nauk SSSR, 210, 727 (1973).
7. A.J.C.Wilson, Nature, 150, 152 (1942).
8. V.I.Andrianov, Z.S.Safina, B.L.Tarnopolsky, Zh.Strukt.Khimii, 12, 1052(1971).
9. G.Germain, P.Main, M.M.Woolfson, Acta Cryst., B26, 274 (1970).