The single crystals have been grown from saturated aqueous solutions at 45°C by slow evaporation of the water. Most of the crystals produced by this method were fairly imperfect. This could be seen by the deviations from optical flatness of the crystal surfaces, these deviations having magnitudes of up to one degree. But it was possible to obtain a few perfect specimens of a size of about 1 cm$^3$.

With such a crystal the $^{23}$Na- and $^{127}$I-NMR spectrum has been investigated as a function of the angle $\theta$ between the magnetic field $H_0$ and the [001]-axis, the crystal being rotated about the [010]-axis. In agreement with the crystal structure of NaIO$_4$ ($\text{space group } C_{4v}^{1} - I4_1/a$, point symmetry of Na: 4, point symmetry of I: 4) the following results have been obtained by a least-squares fit of the experimental curve with the function

$$e^2 q Q(3 \cos^2 \theta - 1 + \eta \sin^2 \theta)/2h$$

(magnetic field $H_0 = 9.34$ kOe corresponding to $r_{\text{Larmor}} = 10.516$ and 7.855 MHz for $^{23}$Na and $^{127}$I, respectively; temperature $23 \pm 2^\circ$C).

![Fig. 1. Angular dependence of the quadrupole splitting $\Delta \nu$ of $^{23}$Na in NaIO$_4$.](image)

- **$\Delta \nu = \nu' - \nu''$**
- $\nu'$ and $\nu''$ are the central-line frequency and the satellite frequencies, respectively.
- $\eta$ is the asymmetry parameter.
- $Q$ is the quadrupole coupling constant.
- $h$ is the Planck's constant.
- $\Delta \nu$ is the frequency difference between the satellites.
- $\eta$ is the asymmetry parameter.
- $Q$ is the quadrupole coupling constant.
- $h$ is the Planck's constant.
- $\theta$ is the angle between the [001]-axis and the magnetic field $H_0$.
- $\Delta \nu$ is the frequency splitting.
- $\eta$ is the asymmetry parameter.
- $Q$ is the quadrupole coupling constant.
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- $\Delta \nu$ is the frequency splitting.
- $\eta$ is the asymmetry parameter.
- $Q$ is the quadrupole coupling constant.
- $h$ is the Planck's constant.
- $\theta$ is the angle between the [001]-axis and the magnetic field $H_0$.
Since there is no correlation between these intensity effects and the angular dependence of the resonance frequencies (a similar maximum in intensity for the $^{23}$Na lines should occur at $\theta = 0^\circ$ and $180^\circ$), the explanation by angular line broadening (mosaic structure) is not possible. This leads us to the supposition that our observation is due to relaxation effects. This observation proves also that one has sometimes to be careful in the interpretation of NMR spectra of powders where it is tacitly assumed that all crystal orientations have the same weight for the averaging.

The obtained value of $e^2q Q/h$ for $^{23}$Na in NaIO$_4$ is much smaller than the corresponding values in NaBF$_4$ ($1008.4 \pm 1.2$ kHz$^1$) and NaClO$_4$ ($836 \pm 20$ kHz$^4$), a fact which is certainly connected with $\eta$ in NaIO$_4$ being zero (NaBF$_4$: $\eta = 0.095 \pm 0.003$), NaClO$_4$: $\eta \leq 0.1^5$). The value of $e^2q Q/h$ for $^{127}$I in NaIO$_4$ must be very large (it should be comparable to $e^2q Q/h$ of Re in KReO$_4$$^5$), showing that the ionic charge distribution in the crystal produces only a minor part of the electrical field gradient at the $^{127}$I-site. The greatest part must be produced by the distortion of the ideally tetrahedral arrangement of the I-O bonds by the surroundings in the crystal.

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Fig. 2. Angular dependence of the frequency $v_c$ of the central resonance line $m = 1/2 \rightarrow -1/2$ of $^{127}$I in NaIO$_4$. $H_0 = 9.34$ kOe. Orientation as in Fig. 1.

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