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# Development of Novel Crystals for Strong Electro-Optic Effect: Rubidium Hydrogen Selenate

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**Abstract:** *The use of highly efficient devices for the treatment of the optical signal continuously drives the research of new optical materials[1-11] for various specific applications. This demand for wideband electro-optic modulators using the pockets effect need high optical quality materials with large electro-optical coefficients and other requirements such as low electric losses and permitivity. Rubidium Hydrogen Selenate (RHSe) Abbreviated name of compound used in the above investigation is put in small bracket which have frequently been used through the paper.Experimental work done to determine purification of RHSe ($RbHSeO_4$) preparation material described in detail, studied crystal growth and effect of growth parameters on the optical quality towards the end. Experimental observations are described in the form of figures and tables.*

**Keywords:** Electro-optic effect, crystal growth, RHSe

## I. INTRODUCTION

Especially interesting for electro-optic and nonlinear optic applicatons,[12-24] the group of the hydrogen-bonded compounds has been largely studied and well known for a long time: especially, the KDP ($KH_2PO_4$) and ADP ($NH_4H_2PO_4$) crystals are widely used in different commercially available system. This latter concerns the first investigation of the electro-optical properties in a single crystal of Rubidium Hydrogen Selenate which is less studied and belongs to the same family as KDP and ADP.

The present study reports a novel technique to achieve very large Electro-optic coefficients, obtained for the first time in this new material, which seems to be promising and may be very interesting for opto-electronic devices, particularly for modulation applications.In addition, the low dielectric permitivity and the weak thermooptic coefficient, are other advantage of this material in regard to requirements for modulators.To address these problems we have chosen for the present study.

## EXPERIMENTAL:

### MATERIALS AND THEIR PURIFICATION:

The chemical compounds were purified and characterised by their melting temperatures before use. The starting material Rubidium hydrogen selenate ($RbHSeO_4$) was prepared by mixing parent components in stoichiometric ratio.

$$Rb_2CO_3 + 2H_2SeO_4 \rightarrow 2RbHSeO_4 + H_2O + CO_2$$

Reactions require very low (less than 2) PH as synthesized material were purified by repeated recrystalization from water solution.

### SET OF THERMOSTAT:

A water thermostat was set up for this investigation. We use a thermostat for controlling the temperature during growth. We measure temperature range 25-40$^0$C auto circulation, self adjustment of temperature, stiring, auto lowering and raising of heating rate was desirable feature.

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**SOLUBILITY MEASUREMENTS:**

Solubility of both components were measured between 25-40$^0$C, water and dionized water are preferred solvents. pH was kept lower than 6.

**CRYSTAL GROWTH:**

The solution growth apparatus as in Fig. 1.1 consist of a sealed glass cylinder of 1.5 lit capacity with two openings. The middle one with an inlet for a seed supporting stirrer, the second one with an inlet for seed, temperature of the bath raised to 40$^0$C supporting stirrer, the second one for introduction of compounds. A saturated solution was prepared and filtered in a large covered vessel[25-28]. A seed was placed to nucleate the crystal. Temperature was lowered in the range of 1-2 degrees per 100 hours for crystal growth.

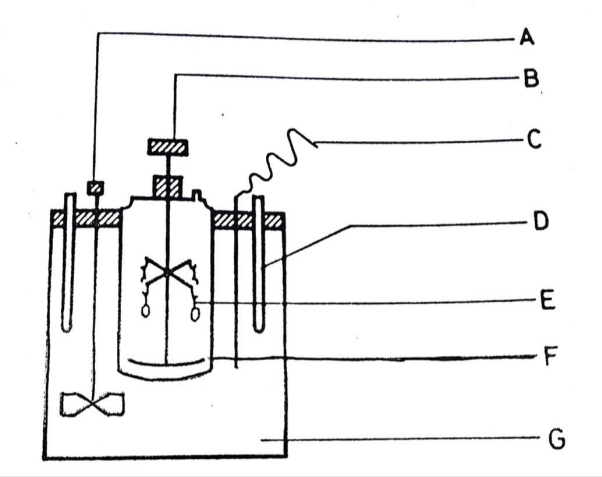


**Fig.1.1 Apparatus used to grow crystals by slow cooling method. A, Motor; B, Seed rotation technique; C, Platinum sensor connected to the regulator; D, Heating by the regulator; E, Seeds; F,Growth solution; G, Thermostated liquid**

**X-RAY CHARACTERIZATION:**

X-ray powder diffraction pattern was used for lattice parameters. X-ray laue was used for determining growth facts 2θ-T(2 Theta-Omega) was used for determing crystal quality. The seeds are then selected after x-ray laue examination, which is, in our opinion, the only valuable test to reveal their crystalline quality. Fig. 1.4 shows nice crystals grown from seeds of good bulk crystalline quality.

Four seeds are attached to the seed carrier by platinum threads of 0.1 mm diameters. The seed undergo alternative accelerated rotations. The duration of a cycle is 2min-2sec. and the maximum speed to be reached is 22 rotation/min.

**OPTICAL CHARACTERIZATION:**

Bulk transparency was determined by polishing the surfaces and observing the transparency with and without wire mesh. Transmittance was measured from 0.3 to 2μm (micro meter) by spectrophotometer. The transmission curve of single crystal determined by Hitachi 340 spectrophotometer is shown in Fig. 1.2

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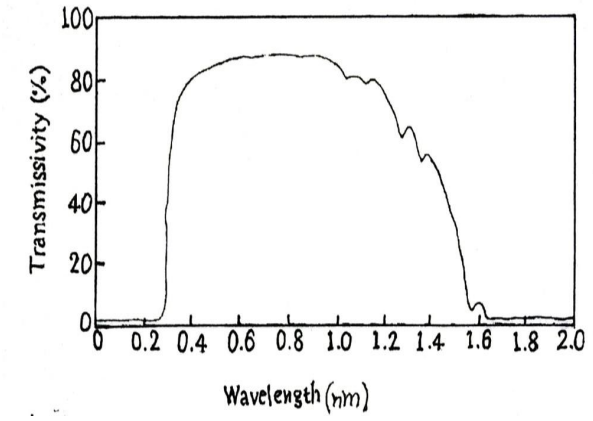


**Fig. 1.2 Tranmission Curve of RHSe single crystal**

**EO MEASUREMENTS:**

Preliminary EO coefficient measurements were made by applying voltage into crystal and measuring the modulation of light.

**RESULTS AND DISCUSSION**

**PURIFICATION:**

Synthesized material was purified by repeating recrystallization from water solution. The source material was obtained from Aldrich Chemical Company. $RbHSeO_4$ was prepared by mixing parent components.

$$Rb_2CO_3 + 2H_2SeO_4 \rightarrow 2RbHSeO_4 + H_2O + CO_2$$

Reaction requires very low (less than 2) pH.

**SET OF THERMOSTAT:**

We use a thermostat for controlling the temperature upto $.01^0C$ during growth. In this thermostate self adjustment of temperature and raising of heating system apparatus used to grow crystals by slow cooling method is immersed in 20 litre water-filled thermostat, also equipped with a stirrer. The temperature of this water bath is controlled by SERELEC electronic regulator with a platinum thermocouple sensor (100 Ω at $0^0C$) providing a regulation rate better than $0.010^0C$ (including the drift regulating from time delays).

**SOLUBILITY MEASUREMENT:**

Solubility of the both components i.e. $Rb_2CO_3$ and $H_2Se_2O_4$ were measured between $25^0C$-$40^0C$. Water and deionized water are preferred solvent. pH was kept lowered than 6.

RHSe solubility increases linearly with temperature within the considered temperature concentration boundary values. The following equations are derived from measurements with corresponding to the temperature in degree Celsius and Y the solubility expressed in grams per litre solution:

- Acetonitrile Y = 202t + 10.69 (A curve in Fig. 1.3)
- Methyl acetate Y= 1.67t - 2.31 (B curve in Fig. 1.3)
- 1,2 Dichloro ethane Y = 1.82t + 11.64 (C curve in Fig. 1.3)

The growth solvent has been selected according to various criterias:

(1) RHSe appears to be soluble in either weak dipolar solvents such as methyl acetate (1.85 D) and 1,2 dichloro ethane (1.75D)or dipolar solvents with as acetonitrile (3.2 D) from Mellan 1970).

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(2) The solvent vapour pressure must be as low as possible within the considered temperature range (typically $25^0$C $28^0$C). This criterion is satisfactory by met as shown by these values.
Acetonitrile 150mm Hg at $35^0$C, 73 mm Hg at $20^0$C
Methyl acetate 284 mm Hg at $35^0$C, 173 mm Hg at $20^0$C
1,2 Dichloroethane 117 mm Hg at $35^0$C, 99 mm Hg at $20^0$C.
(3) The solvent should not be hydroscopic : water may at length interact with the seed surface to the point of inhibiting the growth process. Besides any significant water inclusion would show up in the near infrared spectrum and subsequently increase the absorption on this range in contradiction with applications in view. Therefore, all solvent undergo a thermal treatment in presence of dehydrating substance before distillation.
Five to six days are required to dissolve RHSe in 1.25 litre of solvent and to find the exact equilibrium temperature by adding small quantities of powder until its dissolution stop. (HYVLT 1977).

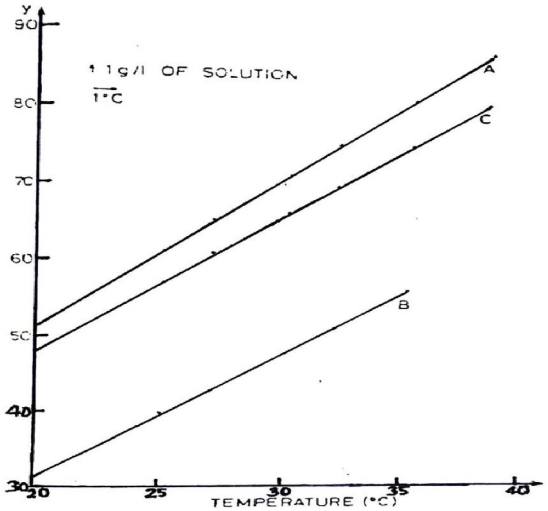


**Fig.1.3 Solubility curves of RHSe as a function of temperature T in three solvents : (A) Acetonitrile, (B) Methyl Acetate, and (C) 1,2- Dichloroethane**

**CRYSTAL GROWTH:**
After dissolution stop, the first seeds are obtained from the evaporation of a saturated RHse solution in the same solvent. Parallelepipedic samples are obtained from single crystal by cutting parallel to the a and c (0.8 x 0.8 $mm^2$), crystallographic exes and cleaving perpendicular to axis b
Rubidium Hydrogen selenate $RbHSeO_4$ crystal presents the triclinic symmetry at room temperature with pseudo-orthorhombic lattice parameters. It possesses ferroelastic, pyroelictric and ferroelectric properties.
The crystals that are being discussed were grown following conditions stated in table 1.1
Solvent evaporation problem was successfully solved. Few preliminary remarks should be make:
(1) For the three different solvent, the initial temperature are not identical but come close (2-$3^0$C deviation)
(2) The overall temperature decrease is variable and depends on the growth progress.
(3) The detailed structure of the sequence of constant-temperature plateau and leaps differs from one solvent to another, but the total duration of the process remains fairly constant. Discontinuous temperature variations may lead to related concentration discontinuities that cannot be accounted for
with time observation scale (around 30 min.) A "solvent effect" on growth seems to be significant. The fusing or pinching this together with the relative orientation of the seed with respect to the thread may cause specific crystalline defects that affect the growth kinetics. Growth speed along axis C is preferential (Table 1.2)

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This corresponds to the smallest dimension of the crystal unit cell. Growth speed along the a and b axes are comparable, which cannot be simply accounted for in view of the very anisotropic cell dimension within the (001) plane.

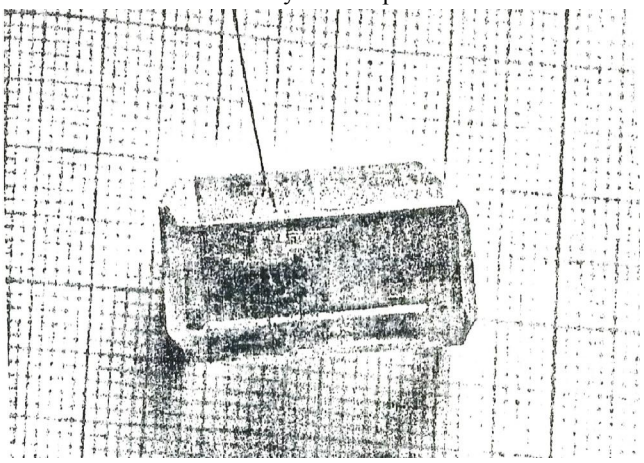

**FIG1.4**

**Table - 1.1**

**Temperature, Concentration and Duration of RHSe and AMA Growth from Solution in Different Solvents**

| Material and Solvent | Temperature, initial (Ti) and final ($T_F$) (*C) | Concentration, initial (Ci) and final (CF) (g/liter solution) | Duration (hr) |
|---|---|---|---|
| RHSe<br>Acetonitrile | $T_i$ = 35.22<br>$T_F$ = 31.25 | $C_i$ = 81.6<br>$C_F$ = 72.67 | 385 |
| Methyl acetate | $T_i$ = 33.44<br>$T_F$ = 28.68 | $C_i$ = 42.26<br>$C_F$ = 40.60 | 337 |
| 1,2-Dichloroethane | $T_i$ = 33.69<br>$T_F$ = 31.40 | $C_i$ = 75.40<br>$C_F$ = 73.78 | 285 |
| AMA<br>1:Ethanol (57),<br>Acetonitrile (43) | $T_i$ = 19.1<br>$T_F$ = 16.4 | $C_i$ = 41.3<br>$C_F$ = 35.7 | 816 |
| II: Isopropanol(50),<br>Methyl ethyl ketone (50) | $T_i$ = 23.8<br>$T_F$ = 20.5 | $C_i$ = 29.1<br>$C_F$ = 22.3 | 792 |
| III: Xylene (70)<br>Acetone (30) | $T_i$ = 29.5<br>$T_F$ = 28.7 | $C_i$ = 91.6<br>$C_F$ = 89.5 | 216 |

**Table - 1.2**

**RHSe Growth Kinetics along the Three Crystallographic Axes in different Solvents: Acetonitrile (Ac), Methyl Acetate (AM) and 1,2-Dichloroethane(DCE)**

| | Axis a | | | Axis b | | | Axis c | | |
|---|---|---|---|---|---|---|---|---|---|
| Crystallographic axes | Ac (1)[a] | AM (2) | DCE (3) | Ac (1) | AM (2) | DCE (3) | Ae (1) | AM (2) | DCE (3) |
| a= 21.359Å | 2[b] | 7 | 5 | 5 | 6 | 6 | 3 | 7 | 5 |
| b=6.111Å | 5 | 4 | 6 | 7 | 6 | 6 | 3 | 6 | 6 |
| c= 5.132Å | 7 | 20 | 10 | 7 | 15 | 15 | 5 | 8 | 15 |

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**Conditions (1) initial temperature 35$^0$C total decrease 286$^0$C, duration 316 hr; (2) initial temperature 35$^0$C, total decrease 4.36$^0$C, duration363 hr; (3) initial temperature 34$^0$C, total decrease 3.50$^0$c, duration 343 hr.**

**X-RAY CHARACTERIZATION**:

The x-ray powder diffraction graph of crystal is showing in Fig. 1.5 and the data of crystal calculated and measured are listed in table-1.3 with four-circle difractometer and solved out the crystal structure. The space groups $P_2/c$, the cell parameters are a = 10.5563 Å, b= 5.6475 Å, c= 12.0482 Å, β= 113.80$^0$. The all crystal structure is shown in Fig. 1.6

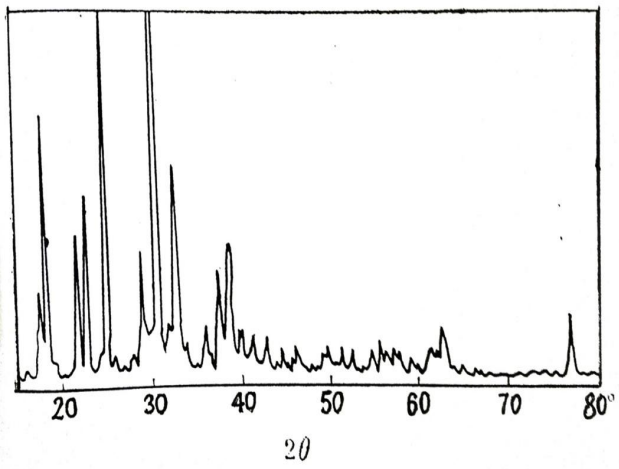


**Fig.1.5 X-Ray Powder diffraction graph of RHSe crystal**

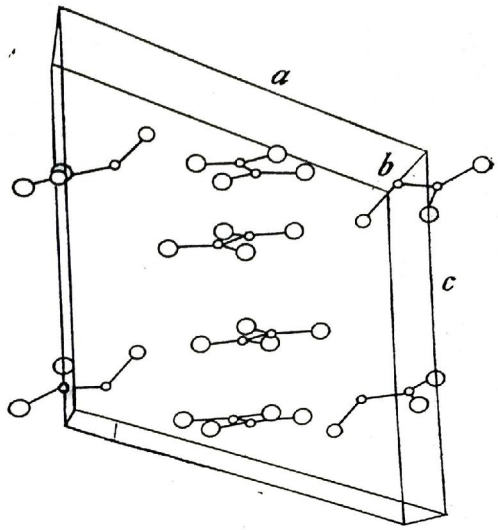


**Fig. 1.6 Structure of RHSe crystal**

**OPTICAL CHARACTERIZATION:**

Light beam is incident along the (100) direction of the sample, whose thickness through which the light posses is 3.7 mm. No transmission on increasing film is coated on surface. The light loss caused by reflections is ignored. It is found that transmission range of the crystal is 0.32-1.35. The highest transmissivity obtained 88%. (Fig. 1.2)

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Major axes of the refractive index are determined by polarized light microscope. $n_y$ is just parallel to c axcis [(001) direction], while $n_x$ is one c-a plane and perpendicular to $n_z$. Three right angle prisms are manufactured. The right angle edge of each prism is parallel to one of three major axes of refractive index. Refractive index of the crystal within visible light range has been measured by V-prism method. Results obtained are listed in table 1.4.

The results suggest that the crystal is negative biaxial crystal.Rubidium hydrogen selenate $RbHSeO_4$ is a crystal which presents the triclinic symmetry at room temperature, with pseudo-orthorhomtric lattice parameters. It possesses ferreoelastic, pyroelectric and ferroelectric properties which have been widely studied.[29-33]

A good optical quality crystal used in our measurement was obtained by isothermal evaporation from aqueous solution and is of the shape of a parallel piped with the dimensions 7.49 x 2.02 x 1.88 $mm^3$ with respect to the pseudo orthorhombic axes. The focus have been polished and electrode with silver paste.

**Table- 1.3: X-ray Powder Diffraction Data of RHSe Crystal**

| 0 | $d_{obs.}$(A) | $d_{cale.}$(A) | hkl | $I_{obs}$ |
|---|---|---|---|---|
| 8.02 | 5.53 | 5.51 | 002 | 2 |
| 8.78 | 5.04 | 5.03 | 011 | 18 |
| 9.13 | 4.85 | 4.84 | 111 | 52 |
| 9.43 | 4.70 | 4.69 | 202 | 2 |
| 10.82 | 4.10 | 4.09 | 112 | 28 |
| 11.23 | 3.955 | 3.944 | 012 | 31 |
| 12.29 | 3.619 | 3.608 | 210 | 76 |
| 12.84 | 3.465 | 3.452 | 302 | 3 |
| 13.34 | 3.338 | 3.331 | 112 | 2 |
| 13.88 | 3.212 | 3.219 | 300 | 3 |
| 14.26 | 3.128 | 3.126 | 303 | 23 |
| 15.03 | 2.969 | 2.9630 | 204 | 100 |
| 16.19 | 2.762 | 2.7559 | 004 | 44 |
| 16.59 | 2.698 | 2.6971 | 212 | 5 |
| 16.93 | 2.645 | 2.6406 | 114 | 4 |
| 17.16 | 2.569 | 2.5654 | 121 | 2 |
| 17.83 | 2.516 | 2.5131 | 022 | 2 |
| 18.53 | 2.423 | 2.4191 | 222 | 23 |
| 19.18 | 2.345 | 2.3447 | 404 | 28 |
| 19.76 | 2.278 | 2.2717 | 213 | 9 |
| 19.98 | 2.254 | 2.2538 | 223 | 7 |
| 20.60 | 2.189 | 2.1857 | 322 | 7 |
| 21.24 | 1.126 | 2.1229 | 320 | 9 |
| 21.79 | 2.075 | 2.0782<br>2.0720 | 123<br>204 | 1 |
| 22.66 | 2.012 | 2.0079<br>2.0074 | 504<br>206 | 1 |
| 23.14 | 1.960 | 1.9623 | 513 | 3 |
| 23.97 | 1.896 | 1.8919<br>1.8915 | 514<br>216 | 2 |
| 24.76 | 1.839 | 1.8373 | 006 | 4 |
| 25.64 | 1.780 | 1.7802 | 515 | 3 |
| 26.11 | 1.750 | 1.7540 | 230 | |

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| | | | | |
|---|---|---|---|---|
| | | 1.7471 | 016 | 3 |
| 27.23 | 1.684 | 1.6844 | 106 | 5 |
| 27.79 | 1.652 | 1.6527 | 332 | 4 |
| 28.24 | 1.628 | 1.6291 | 502 | 2 |
| 28.58 | 1.610 | 1.6098 | 600 | 2 |
| 28.90 | 1.594 | 1.5943<br>1.5935 | 520<br>417 | 1 |
| 29.54 | 1.562 | 1.5625<br>1.5624 | 525<br>331 | 3 |
| 30.72 | 1.509 | 1.5084<br>1.5082 | 521<br>601 | 3 |
| 31.27 | 1.484 | 1.4846 | 430 | 5 |
| 32.46 | 1.435 | 1.4340 | 127 | <1 |
| 33.09 | 1.412 | 1.4119 | 040 | <1 |
| 33.41 | 1.400 | 4.4004<br>1.3996 | 041<br>533 | <1 |
| 34.04 | 1.376 | 13760 | 141 | <1 |
| 34.73 | 1.352 | 1.3519<br>1.3518 | 242<br>431 | <1 |
| 36.89 | 1.284 | 1.2841<br>1.2840 | 814<br>235 | <1 |
| 37.65 | 1.261 | 1.2613<br>1.2609 | 726<br>812 | <1 |
| 38.36 | 1.241 | 1.2406<br>1.2405 | 406<br>616 | 8 |

**Table-1.4**

| λ(Å) / n | 4358 | 4861 | 5461 | 5893 | 6563 | 7065 |
|---|---|---|---|---|---|---|
| $n_x$ | 1.4224 | 1.4191 | 1.4166 | 1.4151 | 1.4135 | 1.4134 |
| $n_y$ | 1.5826 | 1.5759 | 1.5703 | 1.5672 | 1.5634 | 1.5623 |
| $n_z$ | 1.6238 | 1.6204 | 1.6135 | 1.6098 | 1.6053 | 1.6037 |

**ELECTRO-OPTIC MEASUREMENTS (E.O.):**

Electro-optic measurements were performed with the original and very sensitive method, which is based upon the senarmont compensating setup. The optical transmission of such an optical system follow the general law $1 = 1_0 \times \mathrm{Sin}^2 (\Gamma/2 - \beta + \pi/4)$ if the optical absorption is neglected. In this equation $I_o$ and I are respectively the input and output laser intensity and the $\Gamma$ total phase shift introduced by the sample due to the natural birefringence and or its variation as a function of the temperature, the electric field or the strain applied to the sample. Here we are only concerned about the dependence on the electric field.

In this method an ac voltage with a frequency $f$ is superimposed on a dc voltage and we measure the angle $\beta_{2f}$ of the analyzer corresponding to the detection of the output signal with the frequency $2f$.

Measurement are carried out with an electric field applied along t he y axis and a laser beam of 633-nm wavelength propagating along the z axis. They are performed around room temperature with an ac modulating voltage of 150 V peak-to-peak and frequency varying between 100 Hz to 1 MHz, and dc voltage amplitude between - 150 and 300 V.

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Without applying electric field the crystal presents spontaneous birefringence and thus a natural phase shift which needs to be compensated by an appropriate rotation of the analyzer. A clear modulation of the laser beam is achieved with the $RbHSeO_4$ crystal for all available frequencies of the modulated signal which is recorded for a modulation frequency $f$=1 kHz is given in Fig. 1.7, and compared with the corresponding signal obtained with an ADP crystal. The high sensitivity of our method and the large efficiency of the rubidium hydrogen selenate crystal are clearly pointed out in this figure.

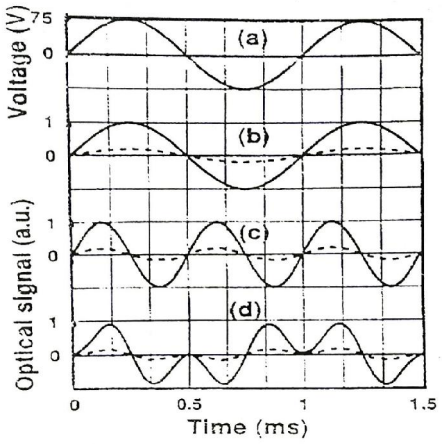


**Fig. 1.7**

The phase shift $F_E = 2x\beta_{2f}$ obtained at T=23.8 $^0$C for a modulation frequency $f$ = 1 kHz is plotted as a function of de voltage in Fig. 1.8. It exhibits a remarkably linear behavior with the applied voltage up to 90 V but deviates from this law for amplitude larger than 100 V and finally saturates. The result is checked to be completely reproducible for various samples. On the other hand, if we compare the values which are obtained for a zero voltage reached with increasing or decreasing amplitude the shift is very large and is equal to $428^0$. This large memory effect is also obtained after switching off the electric field applied to the crystal. Both these phenomena are probably attributable to the domain reversal and require special investigation of the change of birefringence in relation with the domain structure.

Here, we focus our attention on the linear dependence of the phase shift which is consistent with Fig. 1.8, revealing the electro-optic coefficient $r_b$. We can, therefore, deduce from the experimental data and $T_e = (\pi L/ \lambda d) FV$. where d is the interelectrode spacing, L is the crystal length along the laser beam propagation direction, $\lambda$ is the wave length of the laser beam, and F is the figure of merit materials used for modulation application, the figure of merit $F= n^3{}_2 r_b$ of the crystal. We obtain $F= 13.5x10^{-9}$ $mV^{-1}$. The corresponding half-wave voltage inducing a phase shift $T_E = \pi$ is also derived from Eq. 1.1 and found to be equal to $V_\pi = 7V$ for the particular sample used in our experiments. The reduced half-wave voltage characterizing the rubidium hydrogen selenate is obtained when L/d=1: $V^*_\pi = 26.5$ V. The values of F and $V^*_\pi$ obtained for $RbHSe_4$ are given in Table 1.5 and compared with corresponding values reported for the most known electro-optic materials. This comparison shows that rubidium hydrogen selenate is a very promissing material for modulation application owing to its very large F and its very low half-wave voltage. Deviations from the linear dependence of $\Gamma$ vs V which occur for larger voltage can be indeed easily avoided in applications. In addition, to a relatively dielectric permittivity, $RbHSeO_4$ possesses also advantage of a thermo-optic coefficient which is very weak compared to other materials.



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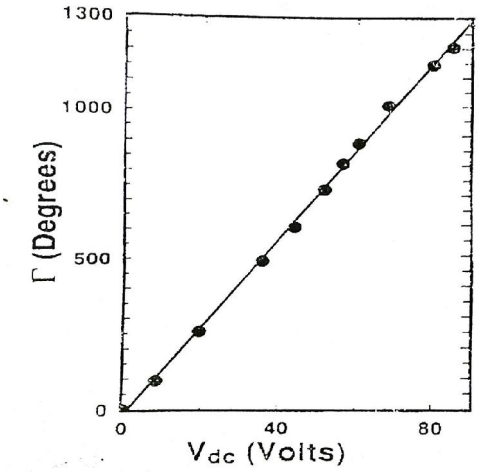


**Fig. 1.8**

**Table-1.5**

| **Compounds** | **F=n$^3$ x r (pm/V)** | **V*(V)** |
|---|---|---|
| $BaTiO_3$ ($r_{42}$) | 24859 | 332 |
| KDP ($r_{63}$) | 40 | 15540 |
| $LiNbO_3$ ($r_e$) | 211 | 2800 |
| KTP ($r_c$) | 184 | 3417 |
| $RbHSeO_4$ | 13540 | 26.5 |

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