Mathematical Model For Estimation of Desublimated Solid Layer Thickness and Surface Temperature

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Abstract:
The thickness of the desublimated solid layer is an important parameter required for evaluation of such desublimation process, especially in the case of solid layers production needed for special applications. A mathematical model has been derived based on the analogy of heat and mass transfer to calculate the solid layer thickness of the desublimated layer during the operation period. The prediction of thickness from the derived model is based on the data related to the applied experimental conditions as well as the physical properties of the vapour-gas mixture inside desublimator. The calculated thickness from the model has been used to calculate the temperature gradient of the solid layer surface during the run period.

Key words:
Desublimation, Solid condensation, Cold trap

Introduction:
Desublimation Process was considered one of the important separation processes used in the chemical plants during the past decades. Desublimation is encountered in modern chemical technology especially in the catalytic oxidation of aromatic hydrocarbon compounds present in the gaseous phase. This technology was frequently applied in the separation of Phthalic and Maleic Anhydrides from the reaction products. As a purification process, desublimation technique has gained an apparent existence in the purification of Salicylic Acid, Benzoic Acid, Anthracene and Naphthalene. Economic studies shows that desublimation technique becomes competitive with distillation, crystallization and solvent extraction. Certain inherent characteristics give it a distinct advantage over other traditional separation methods. Desublimation process has shown a wide attendance in the chemical and petrochemical industries especially in the cases where the distillation technique become non practical to separate a mixture containing components that are very close in boiling points and volatilities. In this case the
number of plates is very large and a large reflux ratio has to be used. Separations of ethylene-ethane mixtures, propylene-propane mixtures, styrene-ethyl benzene mixtures and p-xylene – m-xylene mixtures in producing high grade ethylene, propylene, styrene and p-xylene respectively are good examples (8). The desublimation process had been adopted by many research workers. The desublimator were used to remove TiF4 from the reaction zone in the process of titanium extraction from Ilmenite (9). The condensation of gaseous cyanuric chloride had been achieved using desublimator in the process of cyanuric chloride production method (10). The avoidance of de-hydration step has been achieved using desublimation technique in the process of preparing pyromellitic di-anhydride (11). A process for separation of vaporous heavy metal compounds from a carrier gas wherein the heavy metal compounds are cooled and desublimed and are filtered as particles in a filter (12). Vacuum freezing process was considered an important application in the fields of desublimation technique. Several vacuum freezing processes have been introduced by many workers in the desalination field. These processes contains vacuum freezing vapour compression (CFVC), vacuum freezing vapour absorption (VFVA), Vacuum freezing ejector absorption (VFEA), vacuum freezing solid condensation (VFSC), Absorption freezing vapour compression (AFVC), & Vacuum freezing high pressure ice melting (VFPIM) (13). The vacuum freezing vapour desublimation desublimate vaporization process denoted as (VDDV process) is an improved vacuum freezing process that is useful in separating solvent from solution that contains one or more non volatile solutes (14). There are several cases where the desublimation process is likely to be a suitable separation method (6), most of them are:

1- For unsuitable materials, i.e. temperature or oxidation sensitive at temperatures near to or below the triple point.
2- For distillable material, where its desired to produce the solid product by condensation from vapour phase of a preferable crystal type, crystal size or appearance of the product.
3- For recoverable non volatile and heat sensitive materials required to be separated from volatile material. Freeze drying for the heat sensitive antibiotic, blood plasma are examples for this case.
4- When the material to be separated has a high melting point and at elevated temperature presents many problems such as corrosion of equipment.
5- When the most conventional separation methods of distillation , solvent extraction, adsorption, etc. are either impossible or highly expensive (7).

The Phase Diagram of pure water
The correlation \( T_n = f(p) \) characterizes the surface borders between the liquid (water) and the solid phase (ice) as shown in (Fig. 1) (15).

![Fig. 1: Pressure & Temperature relation of pure water](image)

The three phases (liquid, solid, gaseous) exist simultaneously at the point (0). Such points are called triple point of the substances.
Two opposite phenomena are observed when the system temperature changes, melting $(I \rightarrow II)$ and freezing $(II \rightarrow I)$. The line $A0D$ separates the gaseous phase from the solid and the liquid ones. When the values of the parameters change, the following phenomena are observed:

- Evaporation $(II \rightarrow III)$
- Sublimation $(I \rightarrow III)$
- Liquefying $(III \rightarrow II)$
- Desublimation $(III \rightarrow I)$.

The water and the water vapour have the property to supercool. The dotted line $0B$ expresses the correlation between the temperature and the change of vapour pressure above the supercooled liquid.

**Theoretical Concepts For The Prediction of Solid Layer Thickness and Surface Temperature**

The condensation of saturated vapour from a mixture with inert gas in the conventional desublimator occurred on the inside wall and in the main body of the mixture \(^{(2)}\). Most of the desublimated particles in the main body of the mixture will be pulled or pushed to the inside wall due to the forced action applied inside the desublimator, which is related to the desublimation technique followed. In the first period, the thickness of the desublimated solid layer on the wall is gradually built up due to stable condensation of vapour particles, that will produce a homogeneous solid layer in thickness and dimensions. After some time, the shape of the solid layer will deviate from the homogeneous form, as a result of variables interaction such as that between temperature and pressure in the bulk of desublimator, that will produce unstable system operation.

The form of the homogeneous solid layer and the distribution of temperatures from the bulk of vapour-gas mixture to the bulk of the cooling path is shown in (Figure 2).

![Figure 2: Temperature distribution between the inside bulk of the desublimator and the bulk of water bath](image)

The evaluation of the solid layer thickness and surface temperature is theoretically based on the following assumption in order to simplify the computational procedures.

1. Approximately homogeneous compact circular and cylindrical solid layer without any vacancies in the structure.
2. The heat transfer resistance of the tube wall can be neglected because of a thin wall \(^{(3)}\).
3. The resistance through the gas sub-layer near the solid layer to heat and mass transfer is very small; therefore it can be neglected \(^{(3)}\).
4. The temperature of the desublimated solid on the wall is assumed equal to the wall temperature \(^{(2)}\).
5. The heat and mass transfer process inside the desublimator (cold trap) will be considered as a quasi steady state process.
6. The inlet mixture to the cold trap is saturated and homogeneous, composed of inert gas (Nitrogen) and saturated vapour.
7. The heat transfer rate approached a steady state even while frost continued to accumulate.
8. All condensate settles at the point of formation \(^{(2)}\).
9- The heat and mass transfer process in the axial direction is very small compared with the transfer in the radial direction; therefore, assumed negligible.

10- The vapour pressure of the desublimated solid layer considered very low in comparison with the total pressure of the desublimator (cold trap) (2).

1- Solid layer surface temperature ($T_s$)

The general equation for the circular and cylindrical coordinates, is shown as follows (5):

$$\delta^2 T + \frac{1}{r} \delta T + \frac{1}{r^2} \delta^2 T + \delta^2 T + \frac{Q''}{K} = \frac{1}{\delta t} \ldots (1)$$

For the case of radial transfer, & quasi steady state, equation (1) will be reduced to ordinary differential equation of the form (5):

$$\frac{d^2 T}{dr^2} + \frac{1}{r} \frac{dT}{dr} = 0 \ldots (2)$$

Equation (2) can be integrated twice to obtain:

$$T = C_1 \ln (r) + C_2 \ldots (3)$$

Where $C_1$ & $C_2$ are the Constants of integration

Boundry conditions:
At $r = r_o$, $T = T_w$
Thus:
$$T_w = C_1 \ln (r_o) + C_2 \ldots (4)$$

Note:
($T_f$) will never be equal to ($T_b$), figure (1), unless the inside bulk heat transfer coefficient ($h_i$) is infinitely large, thus an energy transfer from the mixture in the bulk to the solid layer surface will be accomplished by (Newton’s convection law with zero temperature difference); therefore, the rate of heat transfer from the bulk of mixture to the solid layer surface at ($r_f$) must equal the rate of conduction to the solid at ($r_f$). Then;

$$h_i \left( 2 \pi r_f L \right) \left( T_b - T_f \right) = -K_s \left( 2 \pi r_f L \right) \left( \frac{dT}{dr} \right)_{r_f}$$

$$\rightarrow h_i \left( T_b - T_f \right) = -K_s \left( \frac{dT}{dr} \right)_{r_f} \ldots (5)$$

The differentiation of equation (3) gives the following equation:

$$\left( \frac{dT}{dr} \right) = C_1 \ldots (6)$$

Equations (8) and (4) could be solved for $C_1$ & $C_2$ to give the following results:

$$C_1 = \frac{-(T_b - T_w)}{\ln (r_o) + \frac{K_s}{h_i r_f}}$$
$$C_2 = T_w + \frac{(T_b - T_w)}{\ln (r_o) + \frac{K_s}{h_i r_f}}$$

Insertion of $C_1$ & $C_2$ in equation (3) gives an equation for the prediction of temperature as a function of ($r$) for the conduction region between ($r_f$) and ($r_o$) as follows:

$$T = \frac{-(T_b - T_w)}{\ln (r) + \frac{K_s}{h_i r_f}} \ln (r_o)$$
Mathematical Model For Estimation of Desublimated Solid Layer Thickness and Surface Temperature

\[
T_w = \frac{U_a \cdot \Delta T_{lm} + T_{bath}}{h_w} \quad \ldots \quad (13)
\]

Where:
- \( \Delta T_{lm} \) = Logarithmic mean temperature difference inside bulk and cooling bath

\[
\Delta T_{lm} = \frac{T_b - T_{bath}}{\ln \frac{T_b}{T_{bath}}} \quad \ldots \quad (14)
\]

1-2 Estimation of solid layer thickness (b)

The rate of condensate deposit per unit area per mole could be determined by applying a mass balance in accordance with the approach given by Thompson (2), assuming all condensate settles at the point of formation. The mass balance can be shown as follows:

\[
- \frac{V}{dA} \frac{db}{dt} = \rho_s \cdot \frac{db}{dt} \quad \ldots \quad (15)
\]

Equation (15) can be rearranged in the following form by introducing the term \(( \frac{h_i}{h_i} )\):

\[
\frac{db}{dt} = h_{b,i} \cdot M_1 \cdot (-\frac{V}{dA} \frac{dy}{dt}) \quad \ldots \quad (16)
\]

The estimation of the term \(( -\frac{V}{dA} \frac{dy}{dt} )\) will enable the integration of equation (16) and then obtaining an equation for thickness distribution estimate through the time.
For the case of saturated vapour – gas mixture, it is necessary to establish whether or not the mixture will remain saturated. This could be achieved by computing the fraction of condensate which comes out as a mist. If this fraction has a positive value, the gas mixture will tend to remain saturated. The fraction of condensate which appears as a mist could be calculated from the following equation:

\[ \zeta = 1 - \chi_l \]  

\[ \chi \]

Where:
- \( \chi_l \) is the rate of diffusion of condensed vapour across the gas sub layer near the desublimated solid layer per unit area.
- \( \chi \) is the total rate of vapour condensation per unit area.

\( \chi_l \) and \( \chi \) could be evaluated in terms of vapour composition and temperature as follows:

\[ \chi_l = \Gamma . ( p - p_w ) \]

\[ = \Gamma . p . ( \beta ) \]  

\[ \chi \]

Where; \( \beta = p_w / p \)

The relation between heat and mass transfer coefficients had been given by Thompson (2) as follows:

\[ j = \frac{\Gamma . p_r}{( G / M_m ) \left[ \frac{\mu}{(\rho_m \cdot D_v)} \right]} \]

\[ = \frac{h_i}{C_m . G} \left[ \frac{C_m \cdot \mu . D_v}{K} \right] \]  

\[ \chi_l \]

\[ \chi \]

Re-arrangement of equation (19) gives the following form:

\[ \Gamma = \frac{h_i}{M_m . C_m . p_r} \left[ \frac{C_m \cdot \mu . D_v}{K} \right] \]

\[ = \frac{h_i}{M_m . C_m . p_r} \left[ \frac{C_m \cdot \mu . D_v}{K} \right] \]  

\[ \chi_l \]

\[ \chi \]

Substitution of equation (20) in (18) gives the following form:

\[ \chi_l = \frac{h_i}{M_m . C_m \left[ \frac{C_m \cdot \mu . D_v}{K} \right]} \]

\[ \chi \]

In the case of very low vapour pressure of the desublimated solid in comparison with the total pressure of the cold trap, the term \( p / p_r \) could be expressed by the following form (2):

\[ \frac{p}{p_r} = \frac{1}{\ln (1 + y)} \]

Therefore, equation (21) can be converted to the following form:

\[ \chi_l = \frac{h_i}{M_m . C_m \left[ \frac{C_m \cdot \mu . D_v}{K} \right]} \]

\[ \chi \]

\[ \chi \]

The total rate of condensation could be obtained by achieving a material balance over a unit area as follows (2):

\[ \chi = - \frac{v . dy}{d A} \]

Assuming the gas will remain saturated, and the condensable vapour obeys the perfect gas law; thus the term \( dy / d T_b \) could be determined using Modified Clapeyron Equation, which is given by Thompson (2) as follows:

\[ \frac{dy}{dT_b} = \frac{\lambda}{R . T_b^2} \]  

\[ \chi \]

\[ \chi \]

The term \( d T_b / d A \) in equation (23) could be determined by applying a heat balance, i.e., equating the heat transferred
to the inside wall by convection as shown below in (term 1) to the loss in sensible heat of the vapour (term 2) plus the heat of vapour condensing as a mist (term 3), as follows:

\[ h_i \cdot (T_b - T_w) = \] (Term 1)

\[ V \cdot (1 + y) \cdot M_m \cdot C_m \cdot \frac{dT_b}{dA} \]

(Term 2)

\[ + \lambda \cdot \chi \cdot \zeta \]

(Term 3) \[ \ldots (25) \]

Rearranging equation (25) gives the following form:

\[ \frac{dT_b}{dA} = h_i \cdot (T_b - T_w) - \lambda \cdot \chi \cdot \zeta \]

\[ V \cdot (1 + y) \cdot M_m \cdot C_m \]

\[ \ldots (26) \]

Since \( \chi_1 = \chi \cdot (1 - \zeta) \); therefore \( \chi \) could be obtained by substituting \( \chi_1 \) from equation (22) to give the following:

\[ \chi = \frac{h_i}{M_m \cdot C_m \cdot (1 - \zeta)} \left[ \frac{C_m \cdot D_v}{K} \right]^{2/3} \]

\[ \cdot (1 - \beta) \cdot \ln (1 + y) \]

\[ \ldots (27) \]

Substitute equation (24) and equation (26) in equation (23) to obtain the following form:

\[ \chi = \frac{h_i \cdot (T_b - T_w)}{M_m \cdot C_m \cdot (1 - \zeta)} \left[ \frac{C_m \cdot D_v}{K} \right]^{2/3} \]

\[ \cdot (1 - \beta) \cdot \ln (1 + y) \]

\[ \ldots (28) \]

Substitute equation (27) in (28) and solving for \( \zeta \) to give the following form:

\[ \zeta = 1 - \frac{a_1 + a_2}{T_b - T_w + a_1} \]

\[ \ldots (29) \]

Where:

\[ a_1 = \frac{\lambda \cdot \left( \frac{C_m \cdot \rho \cdot D_v}{K} \right)^{2/3}}{M_m \cdot C_m} \]

\[ \cdot (1 - \beta) \cdot \ln (1 + y) \]

\[ a_2 = \frac{R \cdot T_b^2}{\lambda \cdot y} \left( \frac{C_m \cdot \rho \cdot D_v}{K} \right)^{2/3} \]

\[ \cdot (1 - \beta) \cdot \ln (1 + y) \]

Substitute for \( \zeta \) and \( \chi \) in equation (26) to obtain the following:

\[ \frac{dT_b}{dA} = \]

\[ \frac{h_i \cdot (T_b - T_w)}{V \cdot (1 + y) \cdot M_m \cdot C_m \cdot R \cdot T_b^2} \]

\[ + \frac{h_i \cdot (T_b - T_w)}{V \cdot (1 + y) \cdot M_m \cdot C_m} \left( \frac{C_m \cdot \rho \cdot D_v}{K} \right)^{2/3} \]

\[ \cdot (1 - \beta) \cdot \ln (1 + y) \]

\[ \ldots (30) \]

The rate of solid deposit could be evaluated as follows:

\[ -V \cdot \frac{dy}{dT} = -V \cdot \frac{d_y}{dT} \cdot \frac{dT_b}{dA} \]
Substitution of equations (24) and (30) in equation (31) gives
the following :
\[- \frac{V}{h_i} \frac{d y}{d A} = \lambda_i \cdot \frac{d A}{d A} \cdot (T_b - T_w - a_1) \cdot \left( \frac{A}{M_m \cdot C_m \cdot (1 + a_1)} \right) \cdot \frac{1}{a_2} \]
\[\ldots \text{(32)}\]

Substitution of equation (32) in equation (16) gives the following form for Solid
Layer Thickness growing Rate :
\[
\frac{d b}{dt} = \frac{h_i \cdot M_{m} \cdot \lambda}{\rho_s} \cdot \frac{1}{a_1} \cdot (T_b - T_w - a_1) \cdot \left( \frac{A}{M_m \cdot C_m \cdot (1 + a_1)} \right) \cdot \frac{1}{a_2} \]
\[\ldots \text{(33)}\]

Integration of equation (33) gives the following form :
\[b = \frac{h_i \cdot M_{m} \cdot \lambda}{\rho_s} \cdot \frac{1}{a_1} \cdot (T_b - T_w - a_1) \cdot t + C \]
\[\text{Where} ; \]
\[C = \text{Constant of integration} \]
\[\text{Initial condition :} \]
\[at \ t = 0 , \ b = 0 ; \therefore \]
\[b = \frac{h_i \cdot M_{m} \cdot \lambda}{\rho_s} \cdot \frac{1}{a_1} \cdot (T_b - T_w - a_1) \cdot t \]
\[\text{Where} ; \]
\[\ldots \text{(34)}\]

2- Experimental data application :
The application of the derived mathematical model for the experimental conditions related to the work achieved by Ueda \cite{4} has shown a good prediction for solid layer thickness, compared with the thickness measured experimentally. This result has been obtained in the first twenty minutes of run period. The calculated thickness values have deviated from the measured thickness for the period follows the first twenty minutes. This result could be attributed to the tearing of the desublimated solid particles from the surface of solid layer which has occurred due to the forced stream inside the desublimator (Cold trap).
The comparison between the solid layer thickness measured experimentally by Ueda \cite{4} and that calculated from the derived model (based on the experimental conditions related to the work of Ueda \cite{4}) for the individual systems of (Water – Nitrogen) and (Benzene – Helium) are shown in the figures (3) and (4).

Water Vapour + N2

![Graph](image-url)

Figure (3) Comparison between mathematical model prediction and experimental data / Water vapour + N2
The average absolute relative error (AARE) between the measured thickness and predicted thickness from the model for the applied systems related to the work of Ueda (4) are shown in Table (1).

<table>
<thead>
<tr>
<th>System</th>
<th>AARE</th>
<th>First 20 minutes</th>
<th>Total time of run</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water – N2</td>
<td>0.13179</td>
<td>0.14832</td>
<td></td>
</tr>
<tr>
<td>Benzene – He</td>
<td>0.175166</td>
<td>0.17962</td>
<td></td>
</tr>
</tbody>
</table>

The application of the estimated solid layer thickness values for the system of ( Water vapour – Nitrogen ) in equation (34) gives the gradient of solid layer as shown in figure (5).

Fig. 5 shows the desublimated layer surface temperature increase with time up to the limit of quasi constant values, which indicates abstinence of vapour droplets to be condensed because of a forced stream inside the desublimator.

**Conclusion:**
A mathematical model has been developed to estimate the solid layer thickness and surface temperature of the desublimated solid layer inside the bulk of the desublimator based on the analogy of heat and mass transfer. The calculated values for the solid layer thickness from the derived mathematical model has shown a good agreement with experimental data and the thickness measured experimentally related to the work of Ueda (4) for the first 20 minutes of the run period. The deviation in the calculated values occurred for the last period of run. The deviation was attributed to the tearing of the desublimated solid particles from the surface of solid layer which is probably due to the forced stream inside the desublimator (Cold trap). The calculated values of solid layer thickness has been used to diagnosis the gradient of solid layer surface temperature through the period of run.

**Nomenclature**

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Trap heat transfer area</td>
<td>m²</td>
</tr>
<tr>
<td>b</td>
<td>Thickness of deposit</td>
<td>m</td>
</tr>
<tr>
<td>C_m</td>
<td>Specific heat of vapour-gas mixture</td>
<td>J/gm.K⁰</td>
</tr>
<tr>
<td>D_o</td>
<td>Outside diameter of the desublimator</td>
<td>m</td>
</tr>
<tr>
<td>D_v</td>
<td>Binary diffusivity of the</td>
<td></td>
</tr>
</tbody>
</table>
### Mathematical Model For Estimation of Desublimated Solid Layer Thickness and Surface Temperature

**Symbol** | **Description** | **Units**
--- | --- | ---
\( G \) | Mass velocity of the vapour gas mixture | \( \text{gm/m.sec} \)
\( h_i \) | Inside bulk heat transfer coefficient | \( \text{J/m}^2\cdot\text{sec}\cdot\text{K}^\circ \)
\( h_o \) | Outside bulk heat transfer coefficient | \( \text{J/m}^2\cdot\text{sec}\cdot\text{K}^\circ \)
\( K \) | Thermal conductivity of the vapour-gas mixture | \( \text{J/m. sec. K}^\circ \)
\( K_s \) | Thermal conductivity of the solid layer | \( \text{J/m. sec. K}^\circ \)
\( L \) | Length of the cold trap | \( \text{m} \)
\( M_m \) | Mean molecular weight of the vapour-gas mixture | \( \text{gm/gmol} \)
\( M_i \) | Molecular weight of the condensable vapour | \( \text{gm/gmol} \)
\( p \) | Partial pressure of the condensable gas | \( \text{atm.} \)
\( p_v \) | Vapour pressure of the desublimated solid | \( \text{atm.} \)
\( \rho_f \) | Partial pressure of vapour at the interface between the gas and solid phase | \( \text{atm.} \)
\( Q'' \) | Rate of heat generation | \( \text{watt} \)
\( r \) | Location from the center of the cold trap | \( \text{m} \)
\( r_o \) | Outside radius of the cold trap | \( \text{m} \)
\( r_f \) | Location from the outside radius of the cold trap | \( \text{m} \)
\( R \) | Gas constant | \( \text{J/mol . K}^\circ \)
\( t \) | Time | \( \text{minutes} \)
\( T \) | Temperature | \( \text{K}^\circ \)
\( T_e \) | Temperature at the cold trap surface | \( \text{C}^\circ \)
\( T_c \) | Temperature at the entrance to the cooling section | \( \text{C}^\circ \)
\( T_w \) | Tube wall temperature | \( \text{C}^\circ \)
\( T_{bath} \) | Cold water bath temperature | \( \text{C}^\circ \)
\( T_f \) | Temperature at position (f) | \( \text{C}^\circ \)
\( V \) | Flow rate of inert gas | \( \text{gmol / sec} \)
\( y \) | Moles of condensable gas per mole of inert gas | \( \text{gmol / sec} \)
\( \phi \) | Angular direction | |
\( \zeta \) | Fraction of condensate that appears as a mist | |
\( \Gamma \) | Mass transfer coefficient | \( \text{kmol / m}^2\cdot\text{sec}\cdot\text{bar} \)
\( \lambda \) | Latent heat of sublimation per (gmol) | \( \text{J / gmol} \)

Greek letters:

\( \rho \) | Density of the vapour-gas mixture | \( \text{gm / m}^3 \)
\( \rho_m \) | Molar density of solid condensate | \( \text{mol / m}^3 \)
\( \rho_s \) | Density of solid deposit | \( \text{gm / m}^3 \)
\( \rho_v \) | Density of vapour gas mixture | \( \text{gm / m}^3 \)
\( \mu \) | Viscosity of the vapour gas mixture | \( \text{gm / m . sec} \)
\( \Pi = 3.1415927 \)

**References**

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Desublimation, Solid condensation, Cold trap

Figure (5) Gradient of solid layer temperature at position (rf) with time (Experimental data-Ueda (4))

\[
- hj (T_b - T_w) - \lambda \chi \cdot \zeta \chi \cdot \tau = (T_b - T_w) - \Phi_f - \Phi_l - \Phi_h
\]

Figure (6) (Experimental data-Ueda (4))

Keyword: