28TH INTERNATIONAL SCIENTIFIC CONFERENCE OF YOUNG SCIENTISTS AND SPECIALISTS (AYSS-2024)

INDIRECT PROCESS CONTROL USING ELECTRICAL NETWORK PARAMETERS FOR ENHANCED AUTOMATION IN CHEMICAL TECHNOLOGIES

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INTRODUCTION

- **Crystallization in Pharmaceutical Production**
  - Essential for isolating and purifying products.
  - Critical parameters influence product quality and yield.
- **Importance of Control**
  - Parameters like temperature, retention time, and stirring speed impact:
  - Product yield.
  - Content of impurities.
- **Key Factors Influencing Crystallization**

1. *Temperature*
   - Most significant in determining yield and impurity content.
2. *Full Process Time*
   - Greatest impact on yield time.
3. *Stirring Speed*
   - Most significant in determining yield and impurity content.

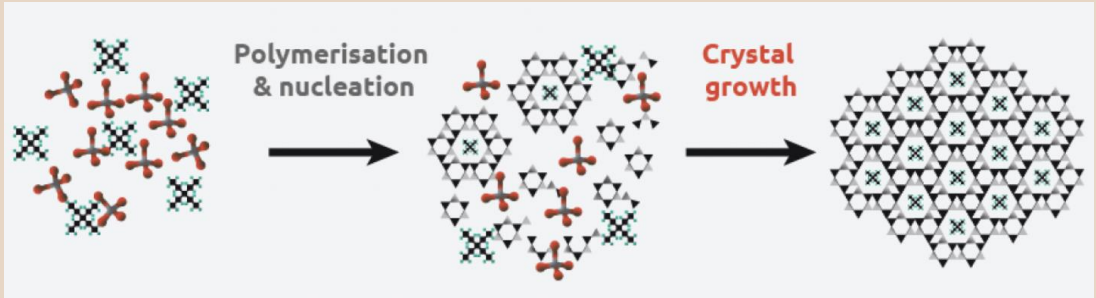
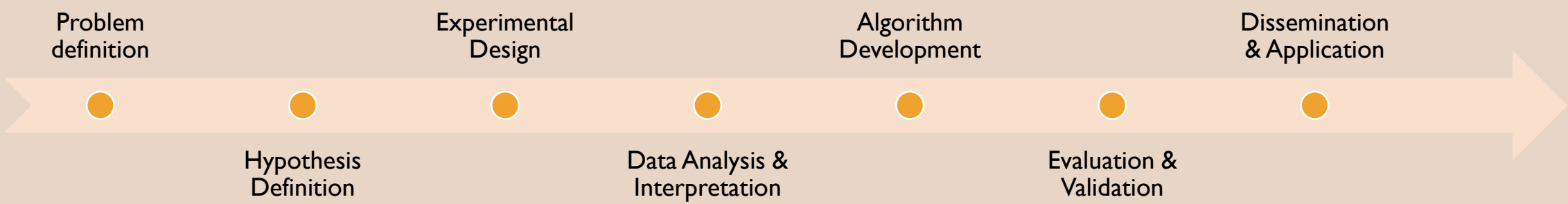


Fig 1. The crystallization process - crystal growth rate vs. nucleation rate. Learn more about Crystallization by Syrris, March 7,2024.

1.Learn more about Crystallization by Syrris,

RESEARCH PIPELINE



PROBLEM STATEMENT

Precisely determining the endpoint of crystallization in pharmaceutical production remains a significant challenge, hindering efficient process control and product quality.

Current Limitations:

- *Lack of Direct Detection:* No readily available methods exist for directly monitoring the end of crystallization.
- *Indirect Parameter Reliance:* Current approaches rely on indirect indicators, which may not be sensitive enough for timely intervention.

Impact of Uncontrolled Crystallization:

- *Product Quality:* Uncontrolled crystallization can lead to variations in crystal size, morphology, and purity, impacting product efficacy.
- *Process Efficiency:* Inaccurate endpoint detection can result in prolonged reaction times, lower yields, and increased production costs.

Goal: Develop a real-time, automated method to accurately determine the endpoint of crystallization, optimizing process control.

TYPICAL METHODS FOR DETERMINING THE END OF CRYSTALLIZATION

- **Defining Endpoint:** Crystallization endpoint is the point where further precipitation is negligible and the process can be stopped without affecting process economics.

Method	Advantages	Disadvantages
Mass Change	- Simple and inexpensive	- Requires accurate mass measurement
	- Sensitive to small changes in mass	- Not suitable for viscous solutions
	- Can be automated	- Susceptible to errors from evaporation
Concentration Change	- Can be used for both dilute and concentrated solutions	- Requires a suitable analytical technique
	- Can be automated	- Can be time-consuming
Viscosity Change	- Sensitive to changes in crystal size and concentration	- Requires specialized equipment
	- Can be used for both dilute and concentrated solutions	- Can be affected by other factors, such as temperature
	- Can be used to monitor crystal growth	- Not as accurate as other methods

PRACTICAL EXAMPLE

- **Challenge:** Consistent product quality requires precise control of crystallization.
- **Problem:** Nucleation detection is difficult due to its microscopic nature.
- **Current Methods:**

1. Optical Sensors
2. Temperature Monitoring.
3. Analytical Sampling.

- **Endpoint Detection**
- **Limitations:** No direct sensors available.
- **Potential Solutions:**

1. ~~Mass Change.~~
2. ~~Concentration Change.~~
3. Mechanical Change.

- **Task:** Develop a reliable method for endpoint detection.
- **Next Steps:** Investigate mechanical change as an indicator.

RESEARCH METHODOLOGY

Experimental Setup:

- *Reactor:* A 200/250 liter anchor stirrer reactor equipped with a temperature sensor.
- *Electrical Monitoring:* OWEN ME210-701 modules for recording three-phase electrical network parameters (voltage, current, power).
- *Frequency Converter:* INNOVERT IVD152B43E for controlling the reactor's stirring speed.
- *Thermostat:* VMT 24 for temperature control.

Process:

- *Target Process:* Synthesis of VM1500A (905420724-2/7) without altering standard parameters.
- *Data Acquisition:* Continuous recording of electrical power parameters (voltage, current, active power, reactive power) at 10-second intervals.

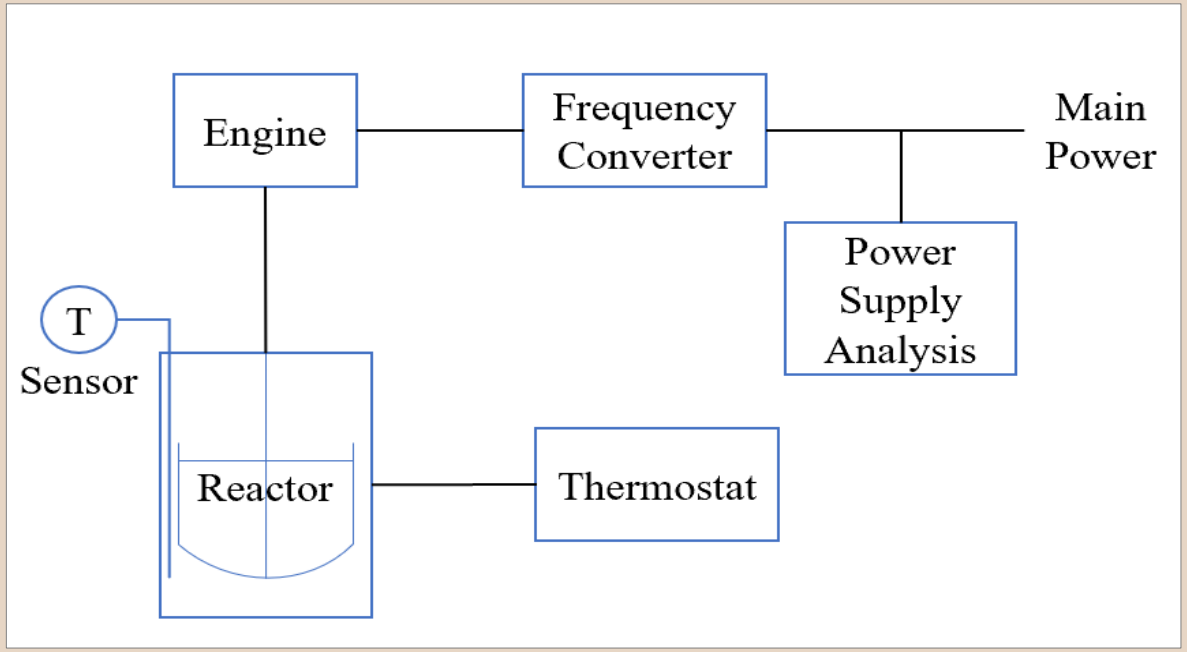


Fig 2. Technical scheme of the experiment.

RESEARCH METHODOLOGY

Measurement Techniques:

- *Voltage Measurement:* Direct connection of voltage measurement inputs to the phases.
- *Current Measurement:* Using current transformers to handle high current values.
- *Data Processing:* Processed using specialized ΣΔ ADC and a microcontroller, with calculations for RMS voltage and current values.

Data Analysis:

- *Power Calculation:* Total, active, and reactive power calculated using standard formulas.
- *Data Transmission:* Data transmitted over an Ethernet network using ModBus TCP protocol.

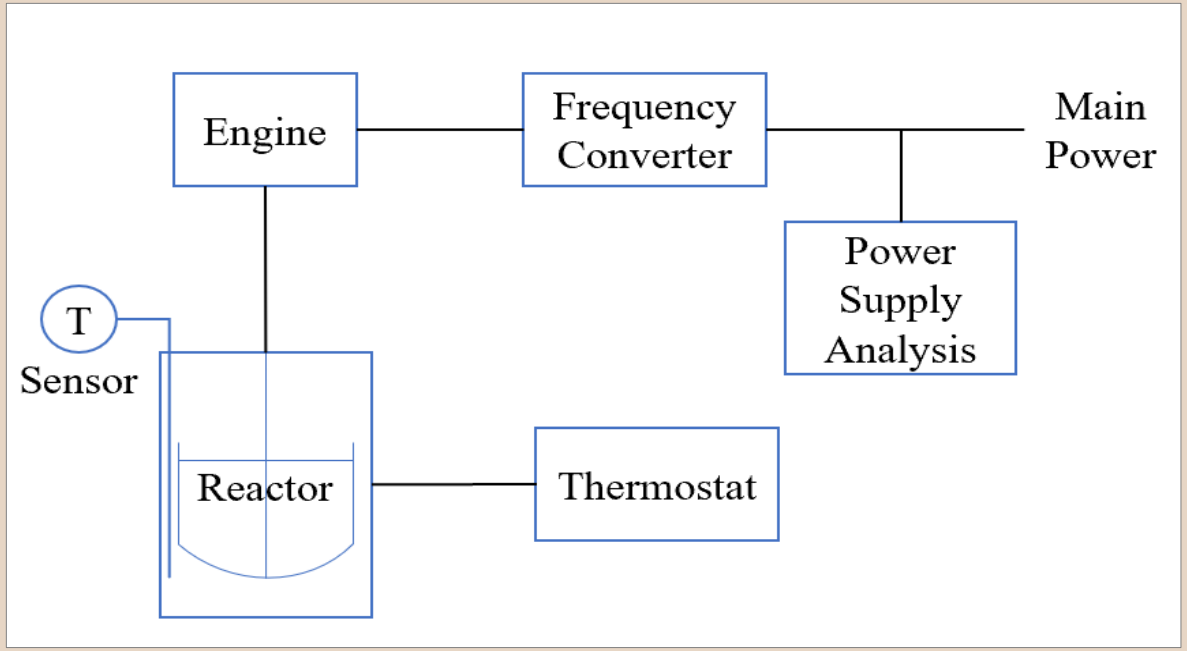


Fig 2. Technical scheme of the experiment.

EXPERIMENT DESCRIPTION

Observation: Active power consumption during crystallization provides a reliable indicator of the crystallization process and potentially its endpoint.

Key Findings:

- *Active Power Increase:* A distinct rise in total active power occurs at the beginning of crystallization, coinciding with the increase in viscosity of the reaction mass.
- *Viscosity Correlation:* Active power is directly related to the viscosity of the reaction mass. As the slurry thickens, more power is required to maintain stirring.
- *Endpoint Indication:* Active power gradually decreases as crystallization progresses and reaches a plateau when equilibrium is attained, indicating the end of the crystallization process.

Data Analysis:

- *Active Power vs. Time:* The most relevant parameter for monitoring crystallization is active power, as shown in Fig3.

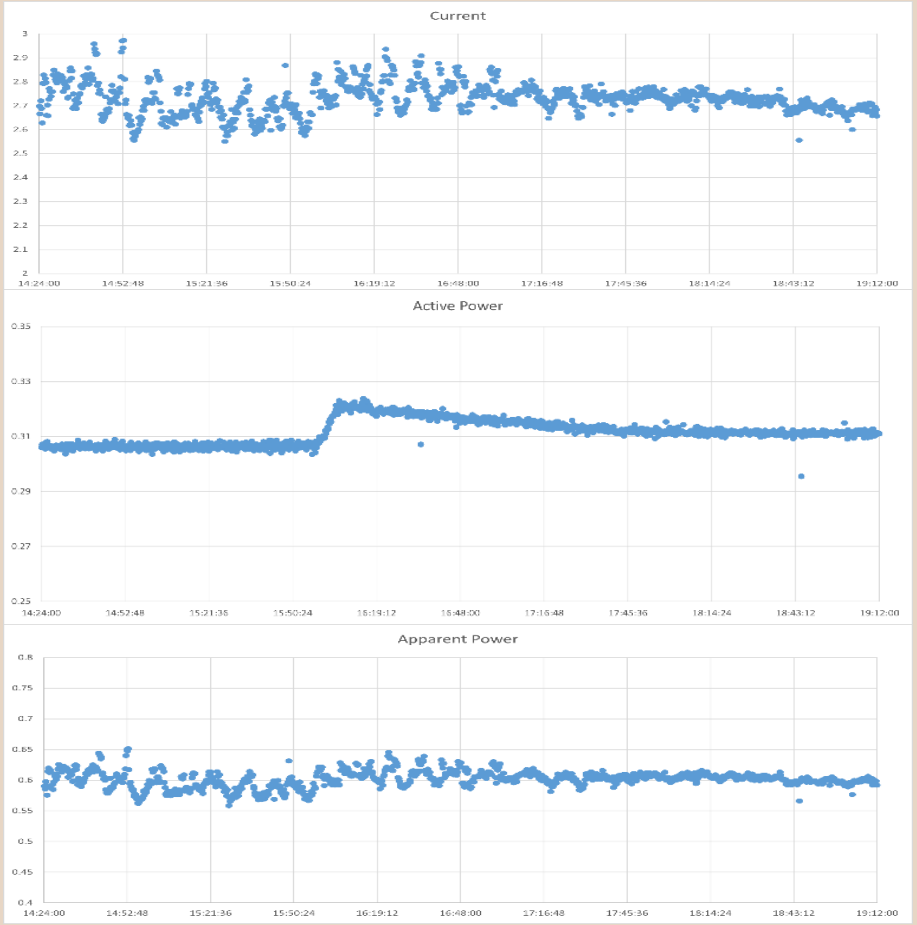


Fig 3. Graphs of the dependence of the current, active power and apparent power on the crystallization time for the product VM1500A 905420724-2/7.

EXPERIMENT DESCRIPTION

Data Analysis:

- *Active Power vs. Temperature:* Figure 4 shows the relationship between active power and temperature, revealing potential influence of exothermic or endothermic reactions.

Mechanism:

- *Viscosity Changes:* The change in viscosity of the reaction mixture affects the load on the stirring motor, directly influencing active power consumption.
- *Exothermic/Endothermic Reactions:* Heat release or absorption can impact active power requirements.

Significance:

- *Indirect Viscosity Measurement :* Active power provides a reliable indirect measure of viscosity changes during crystallization.
- *Endpoint Detection*: The observed decrease in active power can serve as a marker for the completion of the crystallization process.

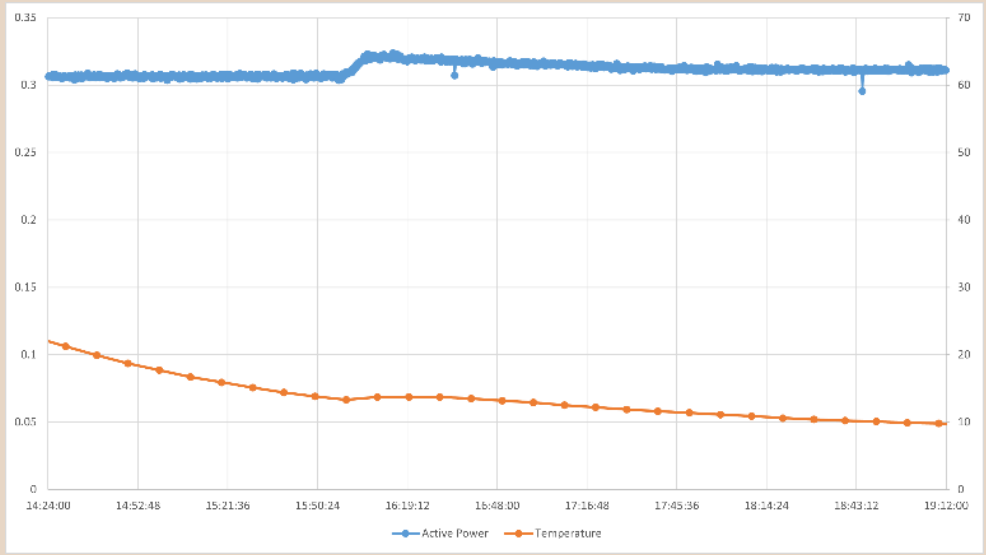
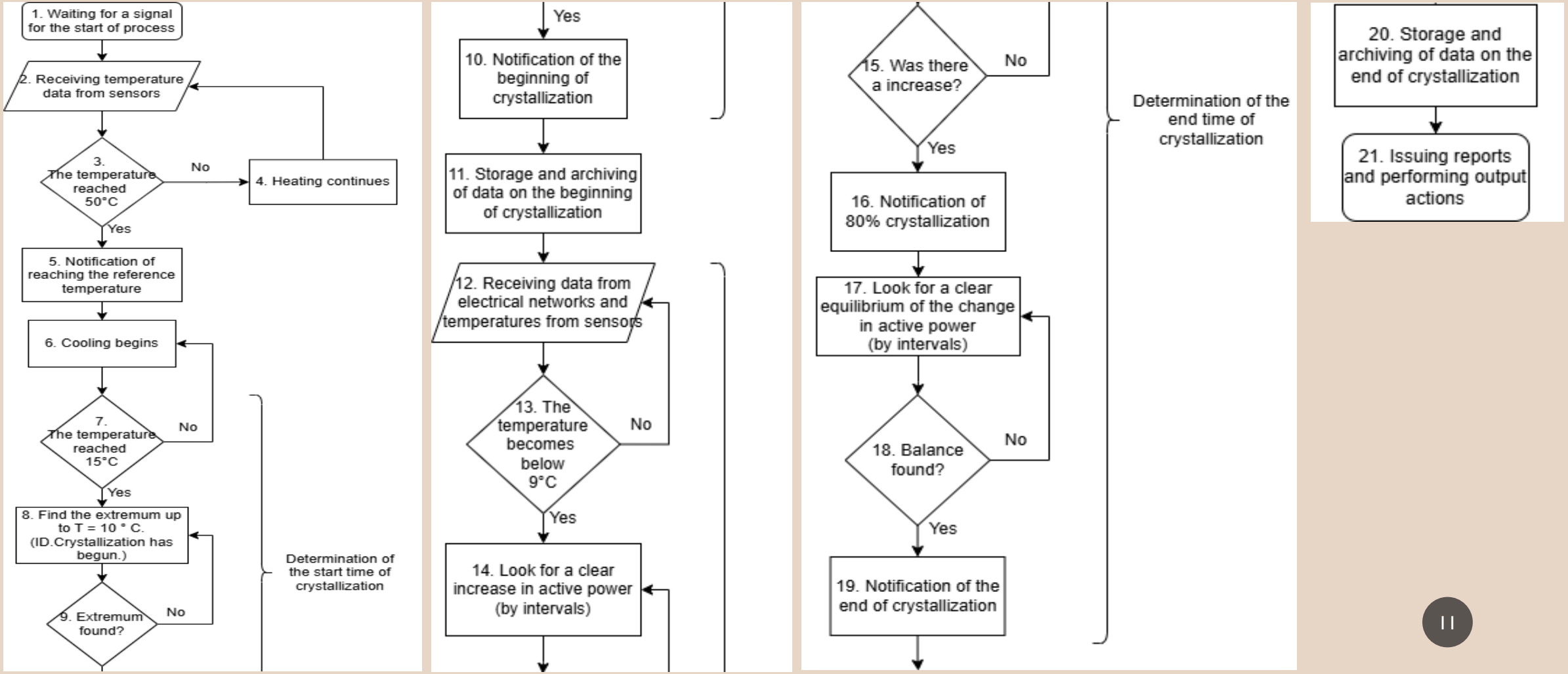


Fig 4. Graphs of the dependence of the active power and temperature with time on the crystallization time for the product VM1500A 905420724-2/7

ALGORITHM DEVELOPMENT



EXPERIMENT LIMITATIONS

- **Challenge:** Indirectly analyzing viscosity changes using electrical power measurements presents limitations due to the need to measure a relatively small change in a large value.

Limitation	Mitigation Strategy
Measurement Accuracy: Small change in a large power value requires high precision.	**OWEN ME210-701:** Cost-effective device with <0.5% error for accurate power measurement.
Interference: External disturbances from the electrical circuit or frequency converter.	**Inductive Filter IEF-7.5/20.5-4:** Filters out harmonics from the frequency converter.
Interference: External disturbances from the electrical network.	**RST020-A Filter:** Protects the measuring device from external network interference.

Advantages of Active Power Analysis:

- *Continuous Monitoring:* Indirectly analyzing viscosity changes using electrical power measurements presents limitations due to the need to measure a relatively small change in a large value.
- *Early Detection:* Sensitive to the initial stages of crystallization, enabling proactive process control.
- *Cost-Effectiveness*: Utilizes readily available and inexpensive electrical instrumentation.

CONCLUSION

Key Findings

- *Viscosity Correlation:* Changes in active power during crystallization reliably reflect changes in reaction mass viscosity.
- *Early Detection:* Active power analysis provides an early indication of sediment formation, allowing for proactive process control.
- *Endpoint Determination:* The endpoint of crystallization can be precisely determined by the stabilization of active power, aligning with traditional validation methods.

Advantages

- *Real-Time Monitoring:* Continuous online monitoring of viscosity changes, enabling accurate control of crystallization parameters.
- *Process Optimization:* Provides insights into crystallization rate and allows for adjustments to temperature, agitation, and seeding for desired crystal properties.
- *Broad Applicability:* Potential for analyzing other industrial processes like dissolution, boiling, and reagent loading.

Superiority of Active Power Analysis

- *Continuous Monitoring:* Provides a dynamic understanding of the chemical system compared to discrete sampling methods.
- *Integrated Control:* Enables comprehensive process control by tracking various physical properties, leading to optimized multi-stage processes.

ACKNOWLEDGMENT

This paper and the research behind it would not have been possible without the exceptional support of my supervisor, Alexey P. Ilyin.

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THANK YOU FOR YOUR ATTENTION

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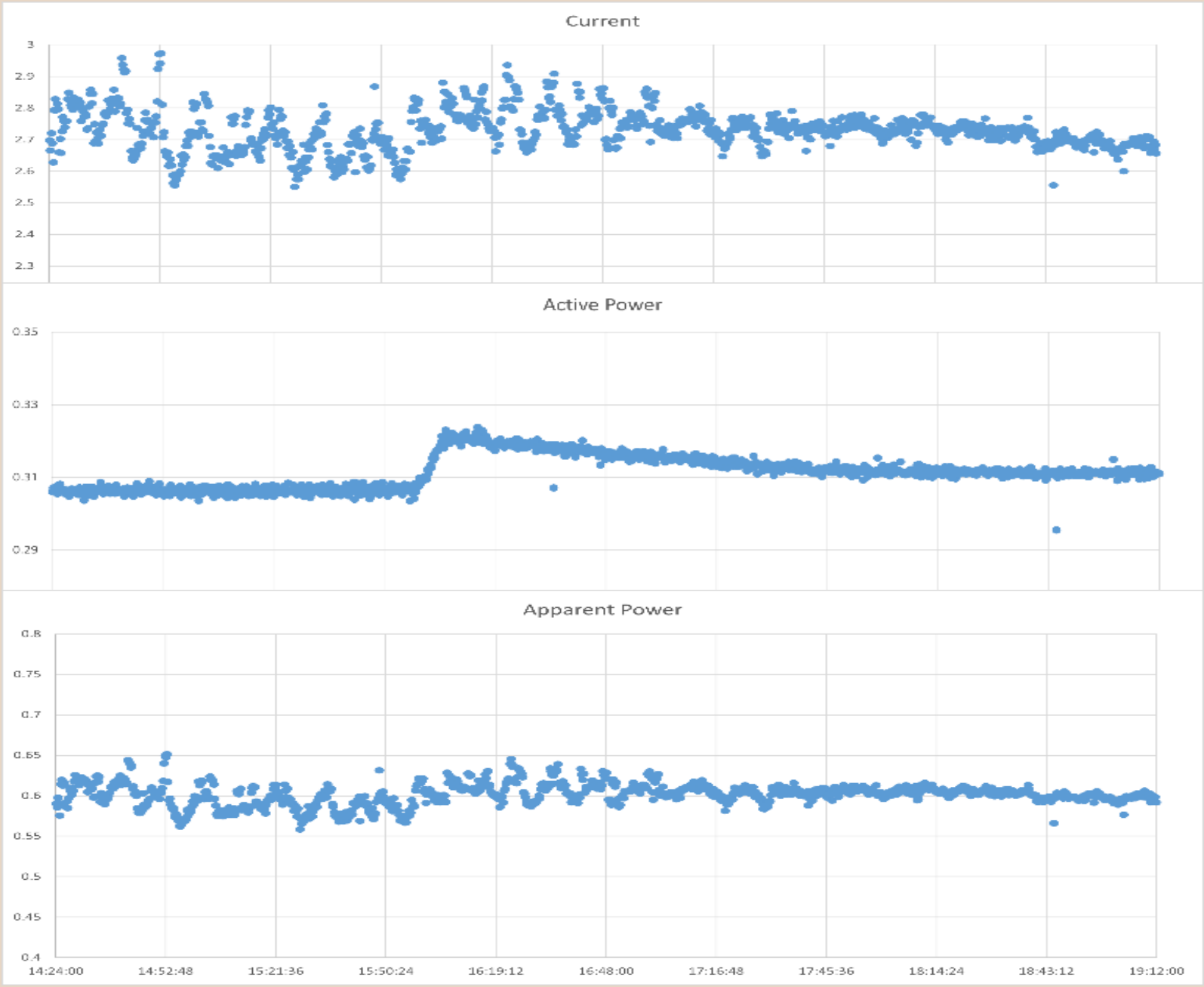
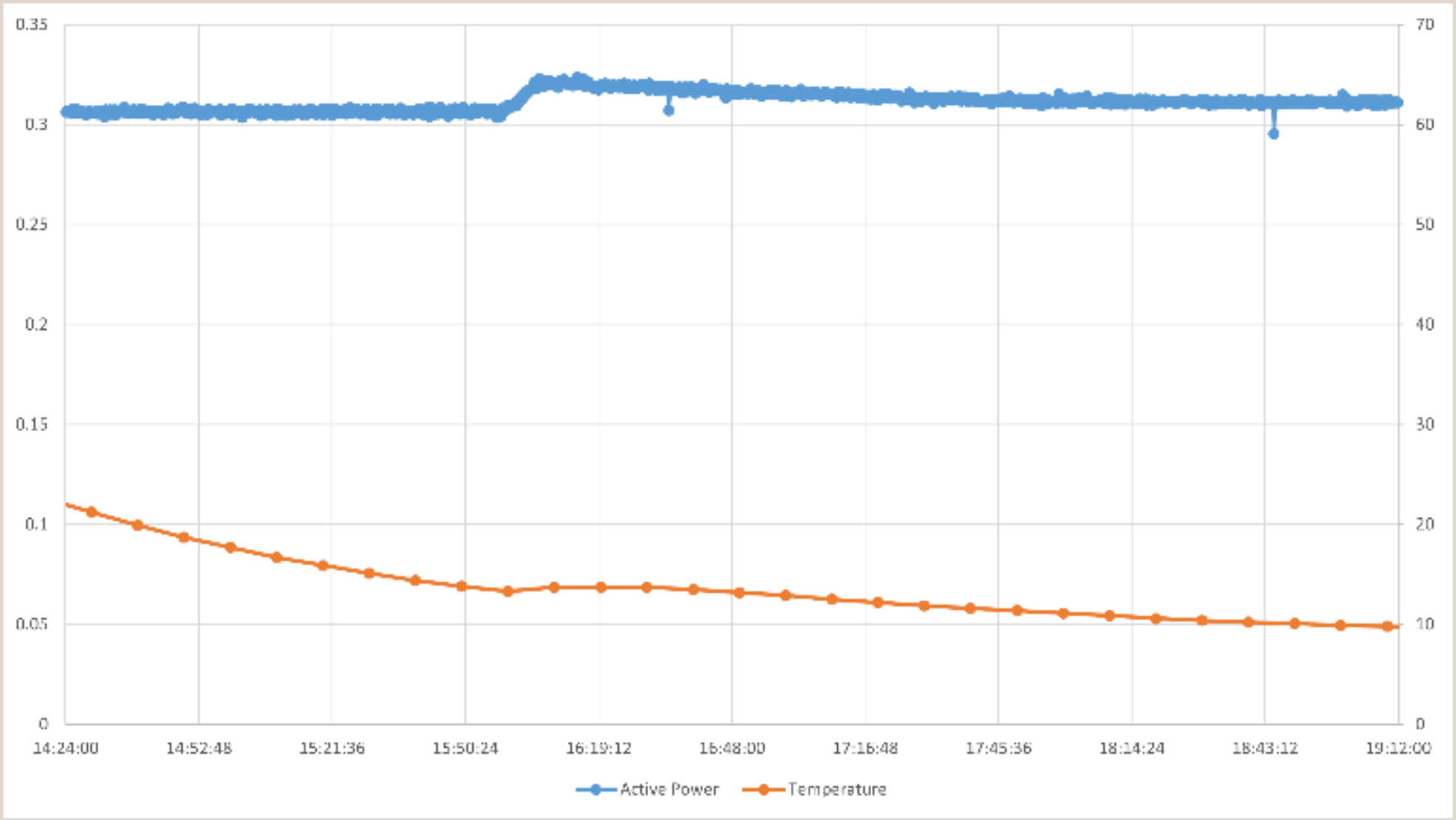
APPENDIX I


Current
3
2.9
2.8
2.7
2.6
2.5
2.4
2.3
Active Power
0.35
0.33
0.31
0.29
Apparent Power
0.8
0.75
0.7
0.65
0.6
0.55
0.5
0.45
0.4
14:24:00
14:52:48
15:21:36
15:50:24
16:19:12
16:48:00
17:16:48
17:45:36
18:14:24
18:43:12
19:12:00

APPENDIX II


0.35
0.3
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0.2
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70
60
50
40
30
20
10
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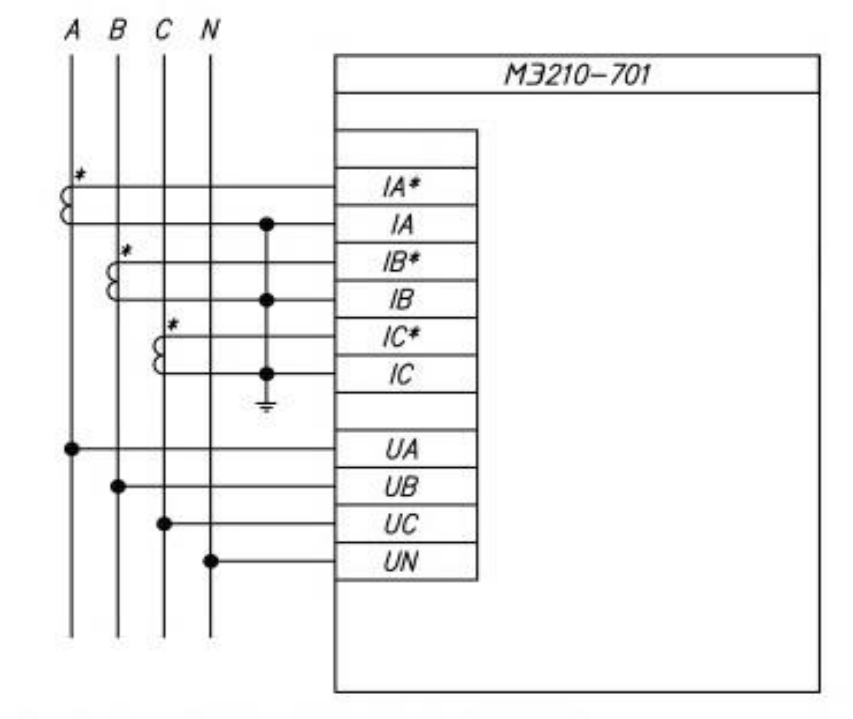
14:24:00
14:52:48
15:21:36
15:50:24
16:19:12
16:48:00
17:16:48
17:45:36
18:14:24
18:43:12
19:12:00
Active Power
Temperature

APPENDIX III



Connection diagram of the OWEN ME 210-701 device.

Subsequent signal processing is performed by a specialized ΣΔ ADC and a microcontroller, where the effective value of the Vrms voltage is calculated using the following formula:

$$V_{rms} = K_V \cdot \sqrt{\frac{1}{T} \cdot \int_0^T V^2(t)dt}$$

where V is the value of the phase voltage, V;
T – period, sec;
KV is the voltage transformation coefficient.

Actual current:
Subsequent signal processing is performed by a specialized ΣΔ ADC and a microcontroller, where the current Irms is calculated using the following formula:

$$I_{rms} = K_I \cdot \sqrt{\frac{1}{T} \cdot \int_0^T I^2(t)dt}$$

where I is the value of the phase current, A;
KI is the current transformation coefficient.

Full power, VA— $S = V_{rms} \cdot I_{rms}$

Active power, VA— $P = V_{rms} \cdot I_{rms} \cdot cos(\phi)$
where φ is the angle of displacement between V and I.
Reactive power, VA— $Q = V_{rms} \cdot I_{rms} \cdot sin(\phi)$
where φ is the angle of displacement between V and I.