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JAPANESE INDUSTRIAL STANDARD

General Rules for Photoelectric
Emission Spectrochemical
Analysis of Metal Materials

JIS Z 2611-1977

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General Rules for Photoelectric
Emission Spectrochemical
Analysis of Metal Materials

Z 2611-1977
(Reaffirmed: 1980)

1. Scope

This Japanese Industrial Standard specifies the following general matters concerning the photoelectric emission spectrochemical analysis of metal materials.

- (1) Apparatus
- (2) Devices and machinery for sampling and preparing samples
- (3) Materials
- (4) Samples
- (5) Methods of preparing samples
- (6) Measuring operation and methods of determination
- (7) Selection and installation of apparatus
- (8) Analysis error and control thereof
- (9) Safety and sanitation
- (10) Items to be described in each standard of analysis method

2. Summary

In this method, the sample is used as electrodes in the state of the metal solid as it is or being suitable formed, or the sample is made a solution and using auxiliary electrodes, and photoelectric emission is produced in the condition adapted to the object of analysis,

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the rays are converted to spectra in a spectroscope, the strength of each analysis line of the element to be determined is measured photoelectrically, and the content of each element in the analysis sample is obtained by using the calibration curves that have been prepared preliminarily based on standard samples.

3. Apparatus

3.1 Summary of Apparatus This apparatus consists of an exciting power source, a light source, a condenser, a spectroscope, a light receiver, and photometer. The exciting power source supply the exciting force for the sample to the light source to cause emission. The condenser condenses and brings the light to the spectroscope. The spectroscope converts the incident light into the spectral lines of the component elements and send them to the light receiver. The photometer photoelectrically measures the intensity of the spectral line of each element that has entered the receiver, and indicates or records the results or converts the measured values into element contents and displays them.

3.2 Constitution of Apparatus The apparatus is constituted of the following units. Fig. 1 shows an example of the constitution.

- (1) Exciting power source
- (2) Light source
- (3) Condenser
- (4) Spectroscope
 - (a) Inlet-slit system
 - (b) Spectral system
 - (c) Outlet-slit system

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(5) Photometer

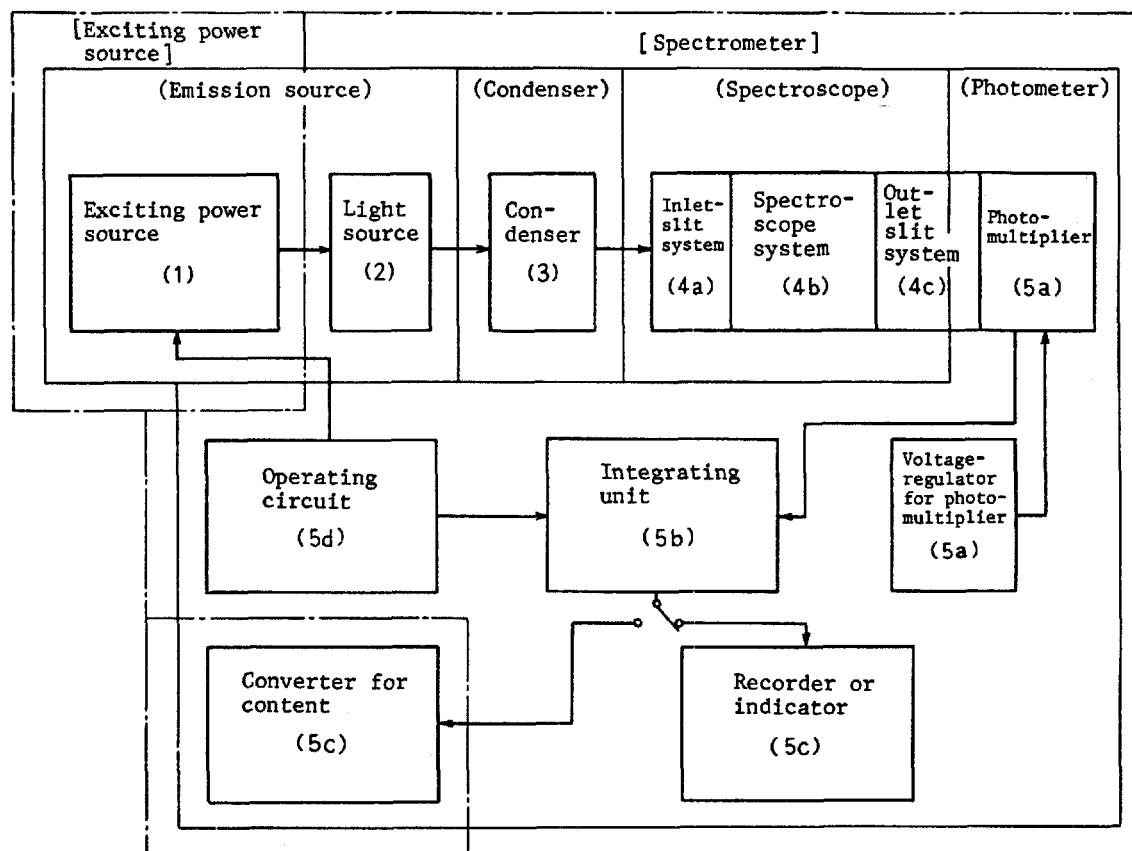
(a) Photomultiplier

(b) Integrating unit

(c) Recorder, indicator and converter for content

(d) Operating circuit

Fig. 1 An Example of Constitution of Emission Spectrochemical Analysis Apparatus



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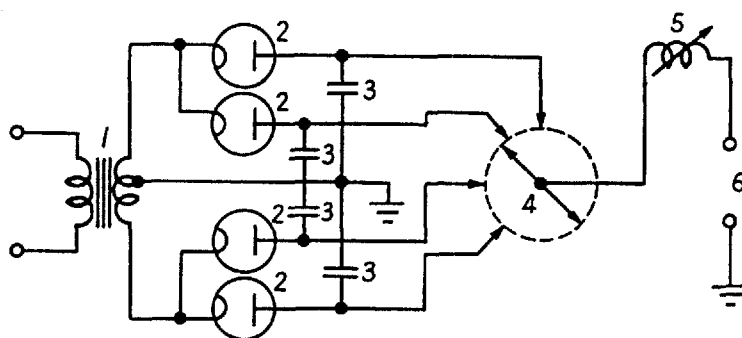
3.2.1 Exciting Power Source This apparatus includes the sparking power source, arcing power source, low-voltage condenser discharging source, etc. These apparatus supply the power source unit for making the sample evaporate and vaporize and exciting it to luminescence. Examples of exciting power sources that are commonly used are described below:

(1) Sparking Power Source

- (a) D.C. High-Voltage Sparking (DC HVS) Power Source This apparatus raises the voltage up to about 10 kV or over by means of a high-voltage transformer, and rectifies this current with rectifier tubes or rectifiers, charge condensers with the rectified current, and then, make the condensers discharge the electricity in turn with a synchronous rotary interrupter.

Fig. 2 shows an example of its main circuit.

Fig. 2 Main Circuit Diagram of D.C. High-Voltage Sparking Power Source



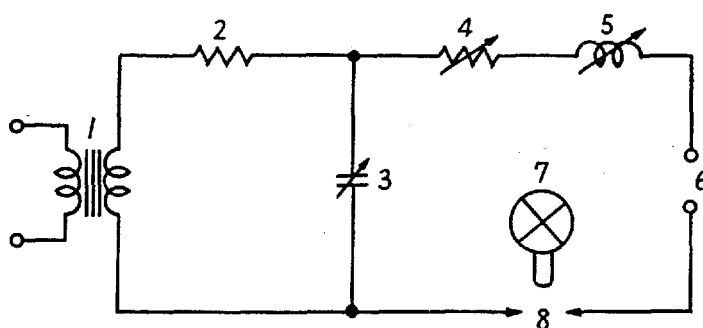
- | | |
|--|---|
| 1. High-voltage transformer:
8 to 15 kV secondary voltage | 4. Synchronous rotary inter-
rupter |
| 2. Rectifier tubes | 5. Inductance coil, 0 to
820 μ H |
| 3. Condenser, 0.007 μ F | 6. Analyzing gap |

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- (b) A.C. High-Voltage Sparking (AC HVS) Power Source This apparatus raises the voltage up to about 10 kV or over with a high-voltage transformer, charges a condenser with this electricity, and controls the discharge from the condenser with the controlling gap that is arranged in series or in parallel with the analyzing gap or with a synchronous rotary interrupter that is arranged in series with the analyzing gap.

Fig. 3 shows an example of the main circuit of the apparatus.

Fig. 3 Main Circuit Diagram of Series-Control-Gap Type A.C. High-Voltage Sparking Power Source



- | | |
|---|--|
| 1. High-voltage transformer:
10 to 20 KV secondary voltage | 5. Inductance coil, 0 to
1250 μ H |
| 2. Resistor | 6. Analyzing gap |
| 3. Condenser, 0.025 to 0.075 μ F | 7. Air-blower |
| 4. Resistor, 0 to 3 Ω | 8. Controlling gap |

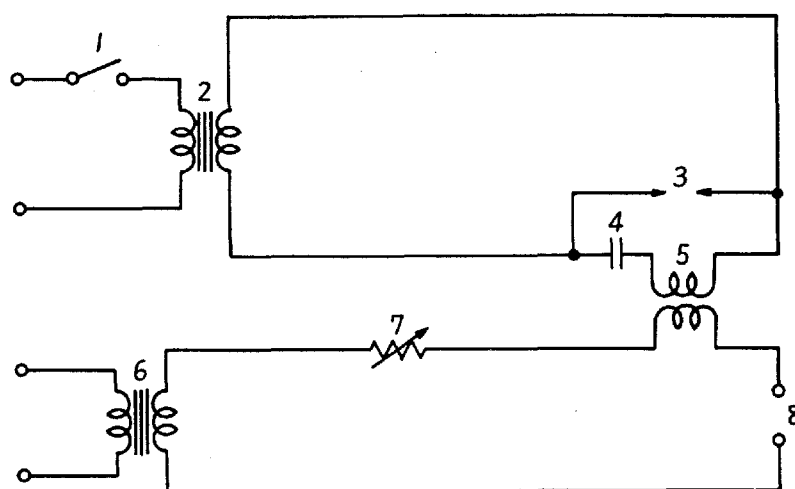
- (2) Arcing Power Source The apparatus in this category includes the d.c. arcing power source and the a.c. arcing power source, of which the latter is chiefly used in the photoelectric spectrochemical analysis. The a.c. arc is of either high-voltage (AC HVC) or low-voltage (AC LVA), and the both are usually ignited by means of high-voltage sparks or high-frequency discharge. In one type of these appara-

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tus, the arcing intervals are controlled by the commercial power frequency unit and in the other type by the second unit.

Figs. 4, 5, 6 and 7 show the examples of main circuits of these apparatus.

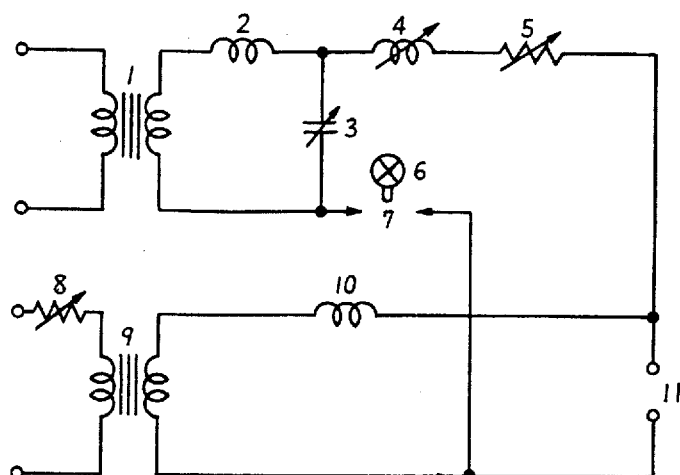
Fig. 4 Main Circuit Diagram of High-Frequency Spark Ignition Type High-Voltage A.C. Arcing Power Source



- | | |
|---|--|
| 1. Time switch for high-frequency ignition | 5. Tesla coil |
| 2. High-voltage transformer: 5 kV secondary voltage | 6. Transformer: 2.4 kV secondary voltage |
| 3. Ignition gap | 7. Resister |
| 4. Condenser | 8. Analyzing gap |

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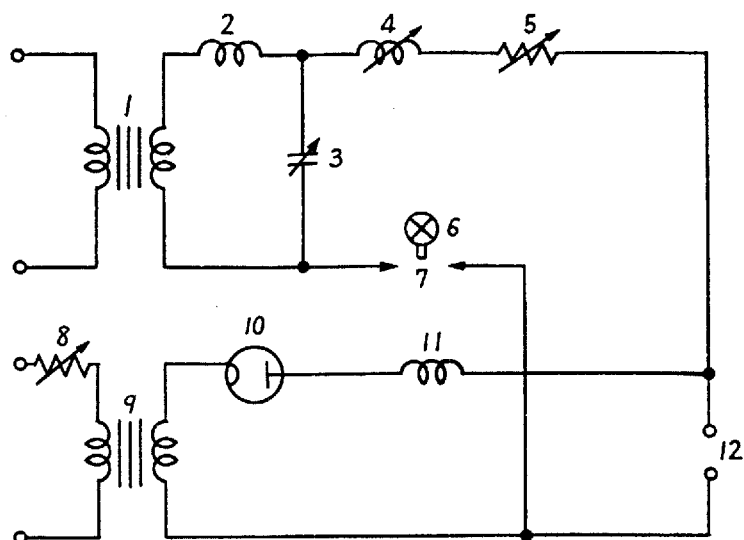
Fig. 5 Main Circuit Diagram of High-Voltage Spark Ignition Type Low-Voltage A.C. Arcing Power Source



- | | |
|--|---|
| 1. High-voltage transformer | 7. Controlling gap |
| 2. Choke coil | 8. Resister, 9.5 to 77 Ω |
| 3. Condenser, 0.0025 to 0.0075 μF | 9. Transformer: 280 V secondary voltage |
| 4. Inductance coil | 10. Choke coil |
| 5. Resister, 0 to 3 Ω | 11. Analyzing gap |
| 6. Blower | |

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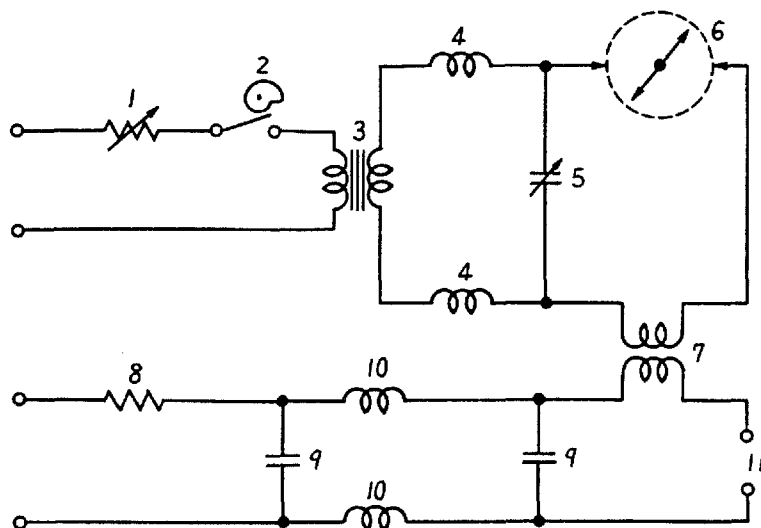
Fig. 6. Main Circuit Diagram of High-Voltage Spark Ignition Type Rectified Arcing Power Source



- | | |
|--|---|
| 1. High-voltage transformer | 7. Controlling gap |
| 2. Choke coil | 8. Resistor, 9.5 to 77 Ω |
| 3. Condenser, 0.0025 to 0.0075 μF | 9. Transformer: 280 V secondary voltage |
| 4. Inductance coil | 10. Rectifier tube |
| 5. Resistor, 0 to 3 Ω | 11. Choke coil |
| 6. Blower | 12. Analyzing gap |

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Fig. 7. Main Circuit Diagram of Pheilsticker Type Interrupted Arcing Power Source

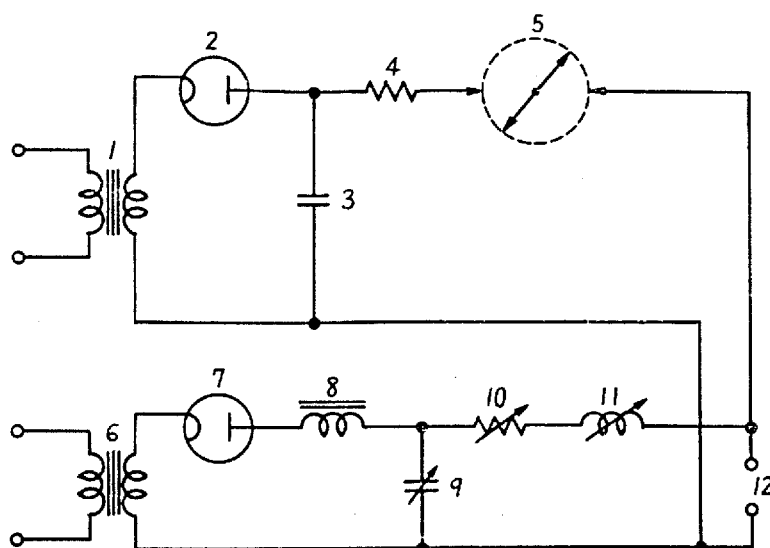


- | | |
|--|-----------------------------------|
| 1. Resister | 6. Synchronous rotary interrupter |
| 2. Interrupter cam (both interrupting ratio and interrupting cycle are variable) | 7. Tesla coil |
| 3. High-voltage transformer: 4.5 to 13.5 kV secondary voltage | 8. Resister, 1 to 80 Ω |
| 4. Choke coil | 9. Condenser |
| 5. Condenser, 0.003 to 0.0066 μF | 10. Choke coil |
| | 11. Analyzing gap |

- (3) Low-Voltage Condenser Discharging Power Source This apparatus is an exciting power source that charges a large capacity condenser up to about 1 kV at most and then, makes ignition with a high-voltage spark. The discharge is controlled by means of a synchronous rotary interrupter, an interrupter with electronic tube circuit, a fixed controlling gap, etc. This apparatus can vary the discharge stepwise from that of a d.c. low-voltage spark (DC LVS) to that of d.c. low-voltage arc (DC LVA). Figs. 8 and 9 show examples of main circuit of this apparatus.

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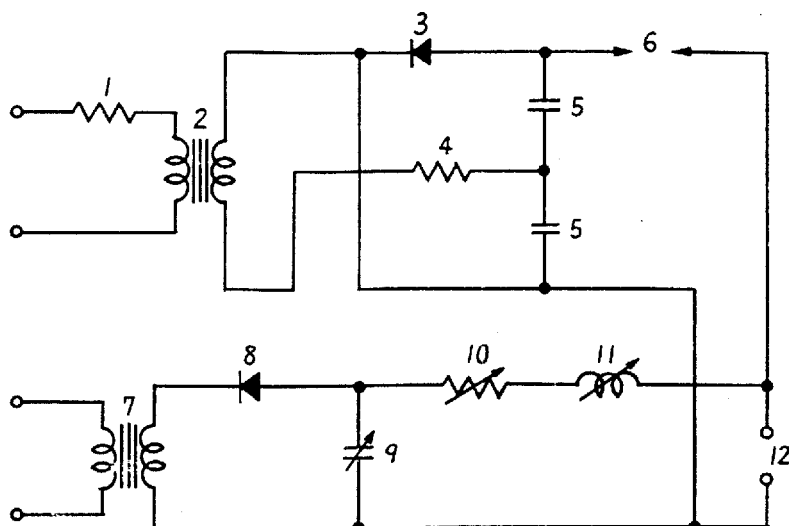
Fig. 8. Main Circuit Diagram of Hasler Type Low-Voltage Condenser Discharging Power Source



- | | |
|---|---|
| 1. High-voltage transformer:
15 kV secondary voltage | 7. Rectifier tube |
| 2. High-voltage rectifier
tube | 8. Choke coil |
| 3. Condenser | 9. Condenser, 2 to 60 μ F |
| 4. Resister | 10. Resister, 2 to 200 Ω |
| 5. Synchronous rotary inter-
rupter | 11. Inductance coil, 50 to
360 μ H |
| 6. Transformer 1 kV:
secondary voltage | 12. Analyzing gap |

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Fig. 9. Main Circuit Diagram of Fixed Control Gap Type Low-Voltage Condenser Discharging Power Source



- | | |
|--|--|
| 1. Resister | 7. Transformer: 1 kV secondary voltage |
| 2. High-voltage transformer: 20 kV secondary voltage | 8. Rectifier |
| 3. High-voltage rectifier | 9. Condenser, 1 to 15 μF |
| 4. Resister | 10. Resister, 1 to 50 Ω |
| 5. Condenser | 11. Inductance coil, 50 or 330 μH |
| 6. Controlling gap | 12. Analyzing gap |

3.2.2 Light Source Apparatus This apparatus is an electrode holder for making the sample emission by electric discharge and is designed to be able to hold the flat sample or bar sample electrode, or an auxiliary electrode for sample and the counter electrode to it. Also it is constructed so as to use a specific gas as the emissive atmosphere, or is designed to rotate the auxiliary electrode for sample, or is designed to facilitate water-cooling of the sample-electrode holder.