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Development of Automatic Plasma Gas Chromatography with Oil-Gas Separation and Gas Enrichment Functions

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Abstract. With the continuous improvement of power grid voltage level, the requirement of equipment condition detection is also increasing. The existing detection technology of dissolved gas in oil is difficult to meet the early monitoring and warning of equipment failure of high voltage level. The technology of oil and gas separation and gas enrichment based on headspace degassing principle was studied. An automatic plasma gas chromatograph with nitrogen as carrier gas was developed. Through integrated design and intelligent control, the instrument realizes automatic analysis of transformer oil. The equipment can reduce the manual operation error and environmental interference in the detection process, with high detection precision and low detection limit. It has a good detection effect on oil samples with low concentration gas content, and has a good application prospect in the analysis and diagnosis of early faults of high voltage oil filling equipment.

Keywords. Gas dissolved in oil, gas separation and enrichment, fully automatic gas chromatograph, plasma detector

1.Introduction

Analysis of dissolved gas in transformer oil is one of the important methods for transformer fault judgment. But testing faces many problems. [1] The chromatographic analysis of dissolved gas in transformer oil is systematic. The analysis process involves oil sampling, degassing, instrument calibration, sample gas qualitative analysis, sample gas quantitative analysis and other links. Each link will bring different degrees of error interference to the final test results. [2,3]

In recent years, with the construction of UHV power grid, more and more high voltage grade transformers or reactors are put into use. How to effectively prevent serious faults of oil filling equipment and ensure the safety of UHV power grid is a major problem faced by the development and operation of power grid. Analysis and detection of characteristic gas in transformer oil is one of the most effective ways to evaluate transformer running condition and carry out equipment fault diagnosis. [4,5] However, the amount of characteristic gas generated by early faults in general equipment is small, and it is dispersed in transformer oil after oil circulation. Some of

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them are adsorbed by insulating paperboard and other materials, resulting in a very low content of gas components in oil (about 10^{-8} V/V level).[6] Limited by the current detection technology, the minimum detection limit of hydrocarbon gases can only reach the level of 10^{-6}L/L, which cannot effectively determine the trace characteristic gases in oil during early faults, and seriously restricts the diagnosis and treatment of early equipment faults. [7,8] The UHV transformer consumes hundreds of tons of oil, which makes the content of characteristic gas components in transformer oil lower, and further improves the difficulty of measuring the characteristic gas components in oil during early fault. [9]

In the process of chromatographic analysis, oil and gas separation is an important step that affects the detection accuracy. The results need to be converted by the coefficient of oil and gas separation to get the concentration of dissolved gas in the oil. [10] However, due to the influence of environmental conditions and personnel operation, the current artificial oil-gas separation method is difficult to ensure the stability and repeatability of degassing between different oil samples. [11,12] It is more difficult to isolate the characteristic gas reliably for the oil samples with less characteristic gas component in the early stage of failure. [13]

On the other hand, in addition to improving the detection performance of the chromatograph itself, by enriching the low concentration gas components and increasing their concentration content, the requirements for the detection performance of the chromatograph are reduced, which is another way to reduce the detection limit of dissolved gas in oil.

In this study, a fully automatic oil-gas separation and gas enrichment device based on the principle of headspace degassing was developed, and a nitrogen-based plasma chromatography was used in parallel. The automatic analysis of transformer oil is realized, which has the advantages of cost saving, good separation, high precision and low detection limit. It provides a better analysis method for the early fault prediction of UHV transformer.

2. Development of Equipment for Gas Separation and Enrichment and Automatic Chromatograph

Using a new dynamic GC headspace method, nitrogen was continuously introduced into the sample, and the dissolved gas component in the oil escaped from the sample with the extraction gas, and then the sample was concentrated through the adsorption device, and finally the sample was desorbed into GC for analysis.

2.1 Development of Oil and Gas Separation Device

Oil and gas separation module (Figure 1), the overall architecture is: power - oil pumping motor -CPU processing unit. The oil circuit part is pumping motor - fixed tubing - headspace degassing chamber - purge and discharge oil. The oil and gas separation process is as follows: the oil sample in the oil needle barrel or the wide mouth bottle is injected into the fixed oil pipe, and the fixed oil pipe is 40ml.When the fixed oil pipe is full, the oil is injected into the headspace degassing chamber. After the headspace degassing, the headspace gas is extracted into the enrichment device, and the oil in the headspace degassing chamber is discharged, waiting for the next injection.

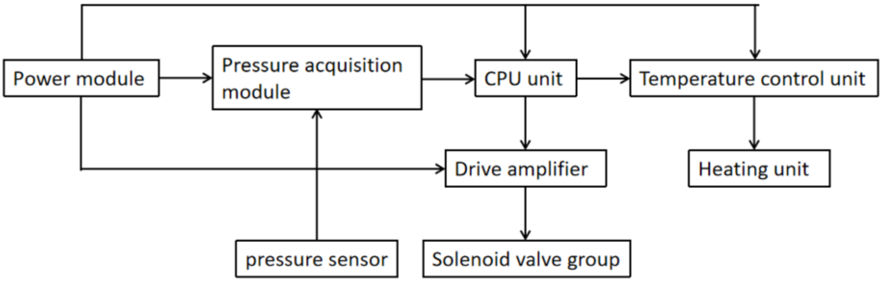

Figure 1. Overall structure diagram of gas separation module

2.2 Development of Enrichment Device

As an independent instrument, the gas flow of the concentrator is redesigned to reduce the use of switching valves and simplify the operation steps of the device (Figure 2). Sample injection can be performed on the two valves, and the device and chromatographic docking can be realized. The gas pipeline and connectors used in this device are all metal materials. The connection between the pipelines is directly welded by card sleeve type hard connection or argon arc welding.

One end of the enrichment unit is a refrigeration module and the other end is an analytical module. In the enriched state, the adsorption tube is enriched at the cold end, and resolved at the hot end during sampling. When the gas in the headspace degassing chamber is enriched by the cold end of the enrichment tube, the enrichment tube switches to the hot end for analysis after the enrichment is completed. After the analysis is complete, the sample enters chromatographic analysis.

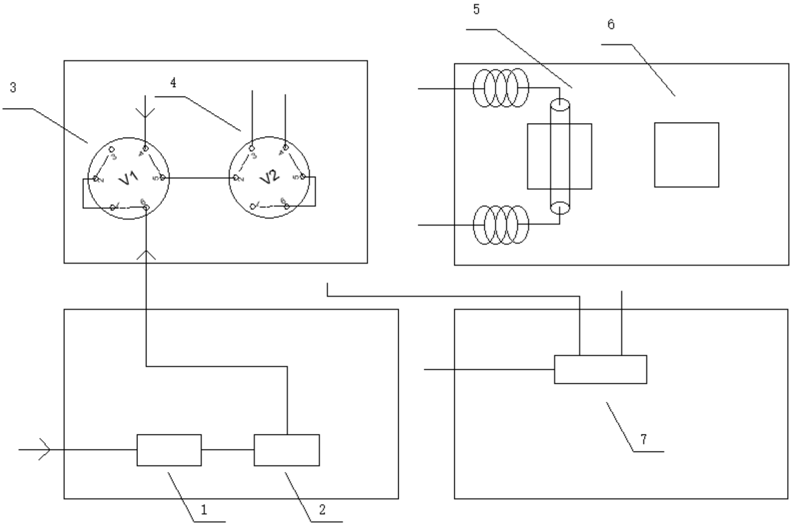

Figure 2. Diagram of connection between oil-gas separation module and concentrator
Attached drawings: 1- fixed tubing, 2- headspace degassing chamber, 3- purge valve, 4- sampling valve, 5- enrichment cold end, 6- enrichment hot end, 7- control system

2.3 Plasma Chromatography Based on Nitrogen as Carrier Gas

Based on the plasma chromatography with nitrogen as the carrier gas, the carrier gas is nitrogen. In order to reduce the detection limit of the instrument in the nitrogen plasma state, the sensitivity of the equipment is increased by vacuum (Figure 3).

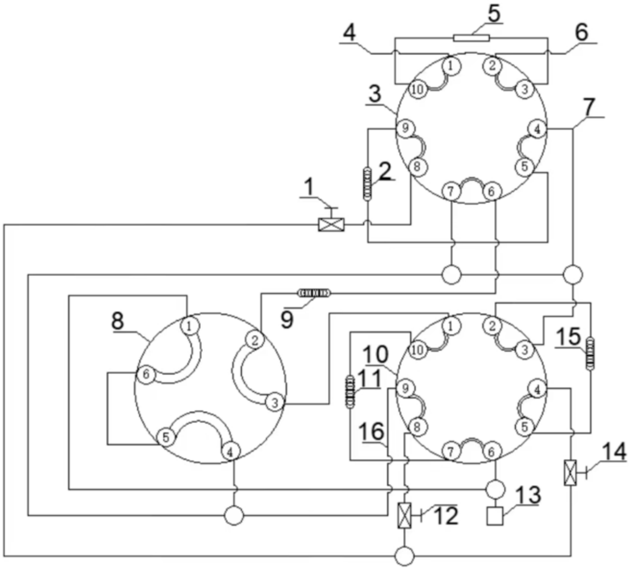

Figure 3. Schematic diagram of plasma chromatography gas path analysis system
Illustration: 1 - First needle valve; 2 - first chromatographic column; 3 - first switching valve; 4-sample inlet; 5 - quantitative ring; 6 - Sample outlet; 7 - Carrier gas, 8 - Second switching valve, 9 - Second column, 10 - Third switching valve, 11 - Third column, 12 - Second needle valve, 13 - Plasma detector, 14 - Third needle valve, 15 - Fourth column, 16 - Carrier gas

3. Overall Unit Performance Test

3.1 Resolution

Chromatographic separation degree, also known as resolution, in order to judge the separation of separated substances in the chromatographic column, expressed by R. The ratio of the difference of the retention time of the adjacent chromatographic peaks to the average width of the two chromatographic peaks represents the separation degree of the adjacent two peaks. The larger the R, the better the separation of the adjacent two components. Generally speaking, when $R<1$, the two peaks partially overlap; When $R=1.0$, the separation degree can reach 98%. When $R=1.5$, the degree of separation can reach 99.7%. $R=1.5$ is usually used as a sign that two adjacent components have been completely separated. When $R=1$, it is called 4σ separation, the two peaks are basically separated, the exposed peak area is 95.4%, and the medial peak base overlaps about 2%. When $R=1.5$, it is called 6σ separation, and the exposed peak area is 99.7%. $R\geqslant1.5$ is called complete separation.

Table 1 shows the peak time and separation degree of components. The separation degree is greater than 1.5, indicating that the method can completely separate each component.

Table 1. Chromatographic peak time and separation

Component	H_2	CO	CO_2	CH_4	C_2H_4	C_2H_6	C_2H_2
Retention time(min)	1.31	6.22	1.92	4.81	2.42	2.82	3.43
Resolution	0	2.49	3.21	3.91	1.92	1.34	1.97

3.2 Concentration Factor

The adsorption performance of the variable temperature concentration device was verified by using 5ppm methane in helium. The peak area of methane detected by chromatography was 1413mV · S. The peak area of 1ml was used as the standard to calculate the actual adsorption multiple of the variable temperature device. The adsorption of 2ml, 5ml, 10ml, 15ml, 20ml and 50ml was found. The adsorption multiple gradually increased with the increase of adsorption amount, but the adsorption capacity reached the maximum after adsorption to a certain volume (Table 2).

Table 2. Adsorption volume and peak area (pA*S)

Adsorption volume (ml)	2	5	10	15	20	50	100
Peak area	593	3145	11514	14619	20992	35831	35676
Actual adsorption multiple	0.42	2.22	8.15	10.34	14.85	25.35	25.24

3.3 Precision

Precision is expressed under the same conditions, the degree of consistency between the repeated measurements of the same sample, is a prerequisite to ensure accuracy, measurable repeatability and reproducibility. Corresponding to two extreme measurement conditions: one represents almost the same measurement condition (called repeatability condition), and the repeatability measures the minimum difference of the measurement results; another representation is to measure the maximum difference in measurement results under completely different conditions (called reproducibility conditions). In addition, the intermediate state condition can also be considered, that is, the intermediate precision condition, which refers to the degree of consistency between the measured values when repeatedly measuring the same amount. Characterize the size of the random error in the measurement process. Chromatographic analysis is a relative measure, so the precision of chromatography is often expressed by repeatability. The repeatability can be expressed by the dispersion of the results of multiple measurements of the same amount under the same conditions. The amount of dispersion of the measurement results, the most commonly used is the experimental relative standard deviation. In the ' JJG-700 chromatographic verification regulation ', the repeatability of the chromatographic analysis method is to calculate the relative standard deviation (RSD) within 5% by seven injections.

Table 3 shows the relative standard deviation of seven samples of each component, RSDS of 10 impurities are all less than 5%, indicating that the repeatability of this method meets the requirements of analysis.

Table 3. Standard gas peak area repeatability of new device (pA*S)

	CH_4	C_2H_4	C_2H_6	C_2H_2	H_2	CO	CO_2
1	11876.032	17668.101	15919.847	7966.664	5381.488	86236.799	368550.765
2	12758.924	19225.884	16222.977	8069.102	4925.096	86254.992	376518.104
3	12540.003	19114.886	16678.525	8632.306	5045.847	89298.257	377643.206
4	12332.163	18793.469	16844.523	8743.567	5112.954	90209.753	376779.366
5	12386.753	18843.549	16984.411	8667.392	5078.843	89961.366	371110.218
6	12139.331	18526.756	16501.428	8618.695	5291.075	89085.505	375237.312
7	12584.524	19042.106	16851.8	8736.95	5205.689	91026.769	381885.353
8	11581.552	17051.505	16147.174	8693.598	5388.555	90931.505	378842.269
9	12457.943	17669.794	15761.316	8222.771	5157.597	88254.172	358587.577
AVG	12295.24	18437.33	16434.66	8483.44	5176.34	89028.78	373906.01
Deviation	371.51	778.44	440.74	307.62	156.19	1804.21	6982.15
RSD	3.021	4.22	2.68	3.62	3.01	2.02	1.86

3.4 Detection Limits of Seven Dissolved Gases in Transformer Oil for New Instruments

Table 4 is the comparison of the theoretical detection limits of the national standard and the nitrogen with oil and gas separation and enrichment functions as the carrier gas plasma discharge detector chromatography, indicating that the newly designed chromatograph is superior to the national standard detection limit.

Table 4. Comparison of the detection limit of national standard and the theoretical detection limit of plasma chromatography

	H_2	CH_4	CO_2	C_2H_4	C_2H_2	C_2H_6	CO
the National standard	≤2	/	≤25	/	≤0.1	/	≤25
Plasma	0.881	0.008	0.5	0.005	0.005	0.005	0.1

4. Conclusions

Because UHV transformer contains more oil, its characteristic gas component content is relatively lower, and the detection limit of dissolved gas in oil is lower, the nitrogen as carrier gas plasma discharge detector chromatography with oil-gas separation and enrichment function is developed. An oil-gas separation unit based on headspace degassing principle is designed. In order to improve the intake efficiency, an enrichment unit is designed. One end of the unit is a refrigeration module and the other end is an analytical module. In the enrichment state, the adsorption tube is enriched at the cold end, and when sampling, the adsorption tube is resolved at the hot end, which can enrich the sample. The plasma chromatography based on nitrogen as carrier gas is used in combination. Nitrogen as carrier gas is easier to obtain, and the cost is more economical than helium. The circuit and software of the equipment are designed, so that the equipment can be integrated control, automatic oil sample analysis is realized, and the original manual error and operation error in mechanical shock method are avoided. After the overall installation of the equipment, the separation degree, precision and detection limit were tested, which is better than the national standard. Nitrogen with oil and gas separation and enrichment function is developed and verified for carrier gas plasma discharge detector chromatography, which enables it to analyze dissolved gas in low concentration and low content oil, and provides a better analysis tool for early fault prediction of UHV transformer.

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References

[1] Kelly, Joseph J. . "Transformer Fault Diagnosis by Dissolved-Gas Analysis." *IEEE Transactions on Industry Applications* IA-16.6(1980):777-782.

[2] Bao-Hua, Dong . "Existent Problems of DGA Application in Transformer Oil and Its Characteristic Gas Identification." *Electrical Equipment* (2008).

[3] Jie, D., Xiao, L., et al. "Study on error sources and solutions of chromatographic analysis of dissolved gas in transformer oil. " *Comprehensive Utilization of Resources in China*, 2020, 38(07),36-40.

[4] Bustamante, Sergio , et al. "Dissolved Gas Analysis Equipment for Online Monitoring of Transformer Oil: A Review." *Sensors* 19.19(2019):4057-.

[5] Haroldo, et al. "A review of monitoring methods for predictive maintenance of electric power transformers based on dissolved gas analysis." *Renewable & Sustainable Energy Reviews* (2015).

[6] He, Huimin , and X. Xu . "Study on transformer oil dissolved gas online monitoring and fault diagnosis method." *International Conference on Condition Monitoring & Diagnosis* 2012.

[7] Mharakurwa, Edwell T. , G. N. Nyakoe , and A. O. Akumu . "Power Transformer Fault Severity Estimation Based on Dissolved Gas Analysis and Energy of Fault Formation Technique." *Journal of Electrical and Computer Engineering* 2019(2019):1-10.

[8] Lelekakis, N. , et al. "Comparison of dissolved gas-in-oil analysis methods using a dissolved gas-in-oil standard." *IEEE Electrical Insulation Magazine* 27.5(2011):29-35.

[9] Gang, Zhang Li . "Relation between the Composition & Contents of Dissolved Gases in Transformer Oil and Insulation Fault Diagnosis of Oil-Filled Power Equipment." *TRANSFORMER* (2000).

[10] Ghoneim, S. et al.. "Adaptive Dynamic Meta-Heuristics for Feature Selection and Classification in Diagnostic Accuracy of Transformer Faults." *IEEE Access*, 9, (2021): 78324–78340.

[11] Muhamad, N. et al. "Comparative Study and Analysis of DGA Methods for Transformer Mineral Oil. " *IEEE Lausanne Power Tech*, (2007) :4538290

[12] Soleimani, et al. "Dissolved Gas Analysis Evaluation in Electric Power Transformers using Conventional Methods a Review." *IEEE transactions on dielectrics and electrical insulation: A publication of the IEEE Dielectrics and Electrical Insulation Society* (2017).

[13] Mohd, S. et al. "Conventional methods of dissolved gas analysis using oil-immersed power transformer for fault diagnosis: A review. " *Electric Power Systems Research*, 216, (2023):109064.