TECHNICAL NOTE

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Comparison of Motor Oils Using High-Temperature Gas Chromatography-Mass Spectrometry

ABSTRACT: The analysis of motor oils has wide applications in the forensic science field from comparing lubricants transferred between an automobile and a victim or crime scene to differentiating the compositions of plastic explosives. In this study, 40 unused motor oils were analyzed and compared by high-temperature gas chromatography-mass spectrometry to determine the potential for oil individualization. Oil samples were also collected from the crankcase dipsticks of 30 cars. Twenty-six of these oils could be differentiated from each other based on visual comparisons of the unresolved envelope (baseline rise due to incomplete separation) and the resolved hydrocarbons in the raw total ion chromatograms (TICs) and smoothed TIC data. Four of these oils were analyzed as unknowns and were correctly related to the corresponding vehicle. The use of extracted ion profiles (EIPs) was explored as a means to further discriminate between the indistinct samples based on the polycyclic aromatic hydrocarbon (PAH) content. The research discussed in this paper demonstrated that differentiation of motor oils was possible by examining the TIC, smoothed TIC, and EIP data.

KEYWORDS: forensic science, high-temperature gas chromatography-mass spectrometry, motor oil, polycyclic aromatic hydrocarbons

The analysis of the base stock in motor oils has many applications in forensic science. They include the possible association of a motor vehicle with a crime victim or scene displaying transferred lubricating fluids, accident reconstruction, arson, and plastic explosive comparisons.

Motor oil base stocks are created from the lubricating fraction of crude oil. This fraction has a very high boiling point $(>400^{\circ}C)$ as well as a high viscosity. The finished motor oil is distilled and refined and can bear little resemblance to the crude fraction from which it was derived (1). In the motor oil industry, the significant characteristic in formulation is the viscosity, which is designated by a Society of Automotive Engineers (SAE) grade. SAE grading assigns low numbers to thin, easily flowing oils, while higher numbers are given to the thick, more viscous oils. Motor oils that have two SAE grades, such as 10W-30, are referred to as multigrade oils. These oils contain polymers that expand as the oil is heated to maintain viscosity (2). A 10W-30 motor oil has a viscosity of SAE grade 10 at low temperatures, but as the engine heats to operating temperatures, the polymers expand, changing the oil viscosity to SAE grade 30.

An analytical technique capable of individualizing motor oils has been of forensic interest for many years. Previously, researchers have shown that motor oils could be differentiated by combining several instrumental techniques, such as infrared spectroscopy, X-ray fluorescence spectroscopy (XRF), gas chromatography (GC), mass spectrometry (MS), and thin-layer chro-

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matography (3). Fluorescence spectroscopy has also been used to discriminate oil samples (4–6). Kubic and colleagues (7,8) used variable separation synchronous excitation fluorescence to individualize motor oils, while more recently, fluorescence quenching was used to demonstrate that the composition of motor oil changes during vehicle use (9). In addition, the analysis of elemental composition has shown potential for comparative purposes (10).

Recently, high-temperature gas chromatography (HTGC) has been used for analysis of oils and other high-molecular-weight hydrocarbons. The reproducibility of the technique as it relates to the analysis of motor oils was addressed by McCormack et al. (11). HTGC was also used in conjunction with XRF to distinguish automobile oil from locomotive oils (12). A combination of HTGC, IR, and scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS) was used to differentiate oil-based lubricants for weapons (13). Roehner et al. (14) used HTGC to compare crude oil samples. HTGC has also been used to distinguish between commercial waxes (15), waxes in artificial fireplace logs (16), and process oils in Composition C-4 (17).

In this study, high-temperature gas chromatography-mass spectrometry (HTGC-MS) with a programmable temperature vaporizer (PTV) injector (18–20) was used to analyze the base stock in automotive oils. Forty unused motor oils and oil samples from the crankcase dipsticks of 30 automobiles were analyzed to determine whether the oils could be differentiated based on the brand, viscosity, and relative age (mileage) of the oil.

Materials and Methods

Samples

Forty unused motor oils were analyzed to create a reference set and to establish a baseline for differentiation. The samples, listed

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in Table 1, consisted of nine brands, 10 SAE grades, four fully synthetic oils, and four partially synthetic blends; however, it was not determined which of the brand names were actual oil manufacturers. Six of the samples were labeled as Mobil Proven Performance 10W-30 but were purchased from multiple locations. Valvoline 20W-50 was present in duplicate, but one was in packaging that was at least 2 years older than the other.

One drop of oil was placed into a 16 mL vial and diluted with trichloroethylene (TCE, Mallinckrodt Baker, Paris, KY) to a mass percentage of c. 0.1% oil in TCE. The solution was then filtered with a Whatman® AUTOVIAL [®] syringeless filter device (Whatman Inc., Clifton, NJ) containing a 0.45 µm polytetrafluoroethylene membrane.

Car Samples

Oil samples were collected from 30 cars by swabbing the crankcase dipstick with a cotton-tipped swab. The swab was then placed in a 16 mL vial and the stick of the swab was broken off to allow for capping. Owing to laboratory supply issues, a replacement for TCE was needed. Through testing in the authors' laboratory, pentane was determined to have equivalent extraction properties as TCE and posed fewer safety concerns. Approximately 4 mL of pentane (Burdick and Jackson, Muskegon, MI)

TABLE 1—List of unused motor oils.

Brand	SAE Grade	Product Name	
Big A	10W-30	All Season	
Castrol	30	Heavy Duty	
Castrol	10W-30	Syntec, Fully Synthetic	
Castrol	15W-40	RX Super	
Castrol	20W-50	GTX	
Castrol	5W-30	GTX	
Castrol	5W-30	Syntec Blend, Partly Synthetic	
Havoline	10W-40	Formula 3	
Kendall	10W-30	GT-1 High Performance	
Kendall	10W-40	Superb 100	
Kendall	15W-40	GT-1 Performance	
Mobil	$0W-20$	Fully Synthetic	
Mobil	10W-30	Proven Performance	
Mobil	10W-30	Proven Performance	
Mobil	10W-30	Proven Performance	
Mobil	10W-30	Proven Performance	
Mobil	10W-30	Proven Performance	
Mobil	10W-30	Proven Performance	
Mobil	10W-40	Proven Performance	
Mobil	15W-50	Advanced Formula, Fully Synthetic	
Pennzoil	50	Aircraft Oil	
Pennzoil	60	GT Performance Racing	
Pennzoil	10W-30	Performax 100, Synthetic	
Pennzoil	10W-30		
Pennzoil	10W-40		
Pennzoil	20W-50	GT Performance	
Pennzoil	5W-30		
Quaker State	10W-30	4 X 4 Synthetic Blend	
Ouaker State	10W-40		
Ouaker State	5W-30	Deluxe	
Trak	30	HD	
Trak	10W-40	Deluxe Quality	
Trak	20W-50	TRX	
Valvoline	10W-30	All-Climate	
Valvoline	10W-40	All-Climate	
Valvoline	10W-40	DuraBlend, Partly Synthetic	
Valvoline	20W-50	Racing	
Valvoline	20W-50	Racing	
Valvoline	5W-30	DuraBlend, Partly Synthetic	
Valvoline	5W-30	All-Climate	

SAE, Society of Automotive Engineers.

was added to the vial to submerge the head of the swab and extract the oil. After extracting for several minutes, the swab was removed and another 4 mL of pentane was added to the vial. The solution was then filtered and analyzed by HTGC-MS. The concentration of the samples was adjusted as needed after the first analysis.

HTGC-MS Analysis

The analysis was conducted on a PerkinElmer (PerkinElmer Inc., Wellesley, MA) Clarus 500 Gas Chromatograph-Mass Spectrometer with a PTV injector. Sample volumes of $2 \mu L$ were injected with a split ratio of 30:1. The injector temperature was ramped ballistically from 50° C to 480° C, held for 2 min, and then ballistically dropped to 400° C and held for 3 min. The column was a bonded phase aluminum clad HT5 (SGE Inc., Austin, TX), $25 \text{ m} \times 0.22 \text{ mm}$ inner diameter, with a film thickness of 0.1 µm. An HT5 column is composed of polycarborane-siloxane, which is equivalent to a 5% phenyl column. The oven temperature was held at 100° C for 1 min, ramped at 15° C per min to 370° C, and then held for 5 min for a total analysis time of 24 min. The transfer line was maintained at 300° C. The hydrogen carrier gas had a flow rate of 1 mL/min. The mass spectrometer had a source temperature of 300° C and was set for a full m/z scan from 40 to 450 in the electron ionization mode. A hydrocarbon standard (D2887 Calibration Mix, Restek, Bellefonte, PA) diluted 1:200 in TCE and a polycyclic aromatic hydrocarbon (PAH) standard containing 16 PAHs in acetonitrile (Polynuclear Aromatic Hydrocarbons Mix, Accu-Standard Inc., New Haven, CT) were used to verify retention times and column resolution and to monitor system performance. The PAH standard was used as received.

Data Analysis Software

TurboMass version 5.0 from PerkinElmer was used for data analysis. Smoothing of the chromatogram allowed for better visualization and comparison of the shape of the unresolved envelopes. The Savitzky–Golay smoothing method was selected with a window size (scans) of ± 99 and a smooth factor of four.

Results and Discussion

Unused Motor Oil References

Initially, a reference library of 40 unused motor oil samples (34 different brands/grades) was created to explore the possibility of individualization. Mobil Proven Performance 10W-30 motor oil was purchased at three different locations (two bottles purchased at each location) but produced indistinguishable total ion chromatograms (TICs) (Fig. 1). Note that a peak, dodecyl acrylate (plasticizer), which appears in the $C_{40}-C_{44}$ range (c. 15 min) in some data, originated from the filter device. Two samples of Valvoline 20W-50 racing oil were in packaging that was at least 2 years apart, but the TICs were also identical. Other oils, such as Valvoline 5W-30 and Pennzoil 5W-30 in Fig. 2, were easily differentiated based on visual comparisons of the resolved hydrocarbons and the symmetry, width, and apex of the unresolved envelope (3). Differences could also be seen in TICs of oils of different viscosities (5W-30, 10W-30, 10W-40, and 20W-50) of the same brand (Fig. 3).

Most synthetic oils, including partially synthetic blends, were easily distinguished from conventional oils. The synthetic oils had more concentrated resolved hydrocarbons and distinct bimodal or multimodal unresolved envelopes (Fig. 4). However, not all

FIG. 1—Total ion chromatograms of two (A and B) of the six Mobil Proven Performance 10W-30 motor oils purchased at different locations.

synthetic oils produced TICs with these unique characteristics. Castrol Syntec 10W-30, a fully synthetic oil, and Castrol 5W-30 Syntec Blend produced TICs similar to conventional oils (Fig. 5).

By visually comparing the envelopes and resolved hydrocarbons, 32 of the 34 oil brands/grades could be differentiated. This led to the possibility that HTGC-MS could identify the oil brand and SAE grade. The analysis of two different brands of oil with different grades, Kendall 10W-30 and Trak 10W-40, demonstrated the complexity of this assignment. The TICs, as shown in Fig. 6, are indistinguishable. Brand identification is complicated, because major motor oil manufacturers produce, bottle, and sell oil under their label and also distribute oil under different brand labels such as those found in discount stores (e.g., Trak Auto). Therefore, associating samples with particular manufacturers was not attempted.

Oils of different SAE grades did show a trend in the shape of the unresolved envelope. As the viscosity of the oils increased, so did the spread and apex retention time of the unresolved envelope.

FIG. 2—Total ion chromatograms of (A) Pennzoil 5W-30 motor oil and (B) Valvoline 5W-30 motor oil.

FIG. 3—Total ion chromatograms of (A) Valvoline 5W-30 motor oil, (B) Valvoline 10W-30 motor oil, (C) Valvoline 10W-40 motor oil, and (D) Valvoline 20W-50 racing motor oil.

This trend was more visible when the peaks in the TIC were removed by smoothing, leaving only the shape of the unresolved envelope. The envelope of the 5W-30 motor oils produced a belllike curve (Fig. 7), which was much narrower than the other grades of oil. The carbon range of the unresolved envelope fell between C_{19} and C_{36} , with the apex of the envelope ranging from C_{24} to C_{28} . The 10W-30 and 10W-40 motor oils, shown in Fig. 8, had similar characteristics and displayed wider envelopes than the 5W-30 oils (Fig. 7). The 10W multi-grade oils generally had slightly longer retention times when compared with the 5W multigrade oils. The carbon ranges for the unresolved envelope and apex of the envelope for the 10W multi-grade oils were from C_{20} to C_{44} and from C_{24} to C_{28} , respectively. Except for one outlier, the 20W-50 oils (Fig. 9), often referred to as racing oil, had even longer retention times than the 10W multi-grade oils. The carbon range of the unresolved envelope was similar to the 10W multigrade oils $(C_{20}-C_{44})$, but the range for the apex of the envelope was much higher, $C_{32}-C_{36}$.

Used Motor Oil Individualization

Once a baseline for comparison was established with the unused motor oils, used motor oils were collected from the crankcase

FIG. 4—Total ion chromatograms of (A) Mobil 1 0W-20 fully synthetic motor oil, (B) Valvoline Durablend 5W-30 synthetic blend motor oil, and (C) Pennzoil Performax 100 10W-30 fully synthetic motor oil.

dipsticks of 30 automobiles to analyze the uniqueness of each. Table 2 shows the make, model, year, and the approximate time since the last oil change of all the vehicles from which oil samples were taken. The vehicles were manufactured between 1985 and 2004 and the miles driven since the last oil change varied from 0

FIG. 5—Total ion chromatograms of (A) Castrol Syntec 10W-30 fully synthetic motor oil, (B) Castrol Syntec Blend 5W-30 synthetic blend motor oil, and (C) Castrol 5W-30 GTX motor oil (conventional).

FIG. 6—Total ion chromatograms of (A) Kendall 10W-30 motor oil and (B) Trak 10W-40 motor oil.

to 12,500 miles. As vehicle oil content was based solely on the owner's knowledge and without a means to verify this information, oil samples from the dipsticks were not compared with the reference collection.

The HTGC-MS analysis of the 30 oils produced 26 unique TICs. Most had similar resolved peaks, but the shapes of the unresolved envelopes were the distinguishing characteristic. An example of this is shown in Fig. 10.

Several of the TICs were initially indistinguishable but distinctions were found upon smoothing. The shape of the smoothed envelope was shown to be reproducible by extracting and analyzing one oil sample seven times. Slight shifts in the beginning, end, and apex retention times of the envelope occurred because of concentration differences and chromatography variations, but the shape did not change. Figure 11 shows an example of two oils that were thought to be the same until the TICs were smoothed and overlaid. The shape of the smoothed envelopes is subtly different and could be considered an envelope retention time shift due to chromatography or concentration. However, this is not the case, as the apex of the two TICs occurs at the same retention time.

FIG. 7—Smoothed total ion chromatograms of all conventional 5W-30 motor oils analyzed.

FIG. 8—Smoothed total ion chromatograms of (A) all conventional 10W-30 motor oils and (B) all conventional 10W-40 motor oils analyzed.

Two of the TICs and smoothed TICs that were indistinguishable were samples taken from BMW cars with comparable mileage (12,000 and 12,500). The smoothed envelopes are shown with the original TICs inset in Fig. 12. Two other vehicles, a 2004 Toyota Matrix and a 1988 Plymouth Sundance, had oil changes on the same day at the same location and produced indistinguishable

FIG. 9—Smoothed total ion chromatograms of all conventional 20W-50 racing motor oils analyzed.

TABLE 2—Make, model, year, time or mileage since last oil change, and oil used in vehicles from which samples were taken.

			Last Oil	
Make	Model	Year	Change	Oil Used
Acura	Legend	1995	55 miles	Unknown
BMW	325i	2003	12,000 miles	BMW 5W-30 Synthetic
BMW	$325 \times i$	2004	12,500 miles	BMW 5W-30 Synthetic
Cadillac	Deville	2000	4000 miles	Mobil 1 Synthetic
	Concourse			
Chevy	Cavalier	1995	1000 miles	Unknown
Chevy	Malibu	2002	2800 miles	Pennzoil 10W-30
Chevy	Malibu	2001	2000 miles	$5W-30$
Dodge	Durango	2002	150 days	Unknown
Ford	Escape	2003	45 days	Unknown
Ford	Explorer	1995	3000 miles	Unknown
Ford	F ₁₅₀	2004	2400 miles	Unknown
Ford	Focus	2002	14 days	Superguard 5W-20
Honda	Accord	2003	120 days	Unknown
Honda	Civic	2002	4 days	Pennzoil 5W-20
Honda	Civic Hybrid	2003	92 days	Mobil 1 0W-20 Fully
				Synthetic
Honda	CR-V	2000	5 days	Unknown
Honda	Prelude	1997	4000 miles	Unknown
Hyundai	Elantra	2004	30 days	Mobil 10W-30
				Synthetic
Hyundai	Elantra	1999	20 days	Pennzoil
Hyundai	Santa Fe	2001	18 days	Pennzoil 5W-30
Mercedes	Diesel 300D	1985	4000 miles	Unknown
Nissan	Altima	2001	960 miles	Castrol GTX 10W-30
Nissan	Frontier	1999	4 days	Unknown
Oldsmobile	Alero	2002	550 miles	Pennzoil 5W-30
Oldsmobile	Cutlass	1992	8 days	Unknown
	Supreme			
Oldsmobile	Intrigue	1998	2500 miles	5W-30
Plymouth	Sundance	1988	1000 miles	5W-30
Toyota	Matrix	2004	3000 miles	5W-30
VW	Passat	2003	3 days	Castrol 5W-30
VW	Touareg	2004	0 miles	Castrol 5W-30

All information is based on the vehicle owner's description.

TICs. The unresolved envelope had the same peaks and comparable shapes even after smoothing.

The concentration of the samples is extremely important when making distinctions in the data and could result in misleading conclusions if not taken into consideration. Samples that are too weak or too concentrated may cause shifts in the unresolved en-

FIG. 10—Smoothed envelopes of oil samples from (A) two vehicles and (B and C) their respective total ion chromatograms.

FIG. 11—Two comparable total ion chromatograms of oil samples from $(A \text{ and } B)$ two vehicles and (C) the smoothed envelopes.

velope beginning, ending, and apex retention times. In extreme cases, the shape of the envelope may be affected, but if the concentrations of the questioned and known samples are comparable, the shape of the smoothed TIC will remain unchanged.

To test the validity of comparing an unknown with a known, four oil samples from the 30 dipsticks were randomly selected and analyzed as unknowns. The data were compared in an attempt to determine the source of each of these four unknowns. Each was correctly associated with the corresponding vehicle after examining the smoothed and unsmoothed TICs.

Extracted Ion Profiles (EIP)

EIPs were examined as a potential for further discrimination between oils with similar smoothed TICs. The most common EIPs (alkanes, cycloalkanes, simple aromatics, and indanes) used in fire debris analysis did not provide additional information, as oils are composed primarily of normal, branched, and cycloparaffins. Any differences seen in these EIPs were already observed in the TICs.

As PAHs are naturally occurring in the environment, especially in petroleum (21), an EIP of common PAHs was created. Table 3

FIG. 12—Two indistinguishable total ion chromatograms of oil samples from (A and B) two BMW cars and (C) their smoothed envelopes.

TABLE 3—List of the m/z ions used in the extracted ion profile and the corresponding PAHs of interest.

mlz	Compound(s) of Interest
178	Anthracene, phenanthrene
202	Fluoranthene, pyrene
228	$\text{Benz}[a]$ anthracene, chrysene
252	Benzo[a]pyrene, benzo[b]fluoranthene, Benzo[k]fluoranthene
276	Benzo[<i>ghi</i>]perylene, Indeno[1,2,3- <i>cd</i>]pyrene
278	$Dibenz[ah]$ anthracene

PAH, polycyclic aromatic hydrocarbon.

lists the m/z ions used in the summed EIP and the corresponding PAHs of interest. When motor oils are exposed to high temperatures, such as those seen in engines, PAH formation will increase (9,21). Unused motor oils showed very little PAH activity (22); thus, the EIP data focused on the samples taken directly from the vehicles. The TIC of the PAH standard is shown in Fig. 13. Not all of the PAHs are represented in the EIP, as the major ions for acenaphthylene, acenaphthene, and fluorene also incorporated unwanted noise from the TIC.

The data presented in Fig. 12 were revisited using the EIP for PAHs. The oils from the BMW vehicles that were originally indistinguishable via the TIC and smoothed TIC data (Fig. 12) began to exhibit some differences in the EIP data (Fig. 14). Oil in both of these vehicles had not been changed for 12,000 miles. While both contain the same PAHs (phenanthrene, fluoranthene, pyrene, and benzo[ghi]perylene), their relative amounts in Fig. 14B are greater than in Fig. 14A. This is significant, as both samples had comparable concentrations. The PAH ratios to other peaks, specifically benzo[ghi]perylene to the unidentified peak on its right, are also noticeably different. It is not yet known whether these data are reproducible; however, an initial examination of the PAH EIP shows potential for discriminating these two samples.

As mentioned previously, the oils from the Toyota Matrix and Plymouth Sundance were changed on the same day at the same place, using the same oil. Between the date of the oil change and the collection of the sample (elapsed time c . 2 months), the Matrix had been driven 3000 miles and the Sundance 1000 miles. These differences are readily seen in the EIP (Fig. 15). Phenanthrene and

FIG. 13—Total ion chromatogram of PAH standard: (1) acenaphthylene, (2) acenaphthene, (3) fluorene, (4) phenanthrene, (5) anthracene, (6) fluoranthene, (7) pyrene, (8) benz[a]anthracene, (9) chrysene, (10) benzo[b or k]fluoranthene, (11) benzo[a]pyrene, (12) indeno[1,2,3-cd]pyrene, (13) dibenz[ah]anthracene, (14) benzo[ghi]perylene.

FIG. 14—Extracted ion profiles of the BMW oils from Fig. 12: (1) phenanthrene, (2) fluoranthene, (3) pyrene, and (4) benzo[ghi]perylene.

pyrene are present in the Matrix oil, but the Sundance sample has only a trace of pyrene.

The idea that PAH formation increases over time and at higher temperatures is also demonstrated in a comparison of two vehicles with oil changes 4 and 5 months before sample collection (Fig. 16). Although details of the exact mileage were not given, it is important to note the additional presence of fluoranthene and benzo[ghi]perylene, both of which were also in the BMW oils. Further evaluation of the data from used motor oils is necessary in order to better understand the relationship between the formation of certain PAHs and the type of motor oil used, the vehicle, and its operating conditions.

Future Research

In future studies, the variation in oil over time from one vehicle will be examined. The authors expect the chromatographic profile and the PAH content of the oil to change as it cycles through the engine repeatedly for several thousand miles. Research will be

FIG. 15—Extracted ion profiles of (A) 1988 Plymouth Sundance and (B) 2004 Toyota Matrix showing the presence of (1) phenanthrene and (2) pyrene.

FIG. 16—Extracted ion profiles of oil from vehicles changed (A) 4 months before collection and (B) 5 months before collection. Labeled peaks are (1) phenanthrene, (2) fluoranthene, (3) pyrene, and (4) benzo[ghi]perylene.

conducted to monitor the oil and determine what differences occur and what the significance of these differences is.

Conclusions

HTGC-MS was used to analyze and compare 40 unused motor oils comprising a variety of different brands and grades. By examining the unresolved envelope, the resolved hydrocarbons, and the smoothed TIC, 32 of the 34 different oil brands and grades were distinguishable from each other. While labeled brand and labeled SAE-grade identification were not possible due to complicated manufacturing and distribution processes, there was a trend in the shape of the unresolved envelopes. As the viscosity of the oils increased from 5W to 10W to 20W, the retention times lengthened and the envelopes broadened. Unused oils of the same brand and viscosity, but from different stores, could not be differentiated.

Oil samples were collected from the dipsticks of 30 vehicles and produced 26 distinct TICs. Only by smoothing the TICs and comparing the shapes of the unresolved envelopes were several of these chromatograms differentiated from one another. Two of the indistinguishable TICs were samples taken from BMW vehicles, which contained synthetic oil supplied by the BMW dealers and were driven approximately the same distance. Examination of a summed PAH EIP for used motor oils indicates the potential for further discrimination.

Overall, HTGC-MS is a valuable comparative technique for the analysis of motor oils. Trends in oil viscosity and changes in the PAH content may provide additional information when comparing questioned and known samples. Examination of the TICs, EIPs, and smoothed data showed a high potential for discrimination of motor oil samples.

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