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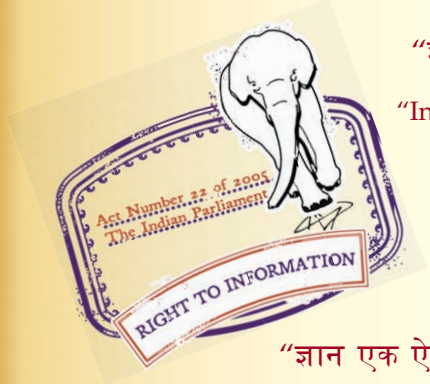
IS 5182-12 (2004): Methods for measurement of air pollution, Part 12: Polynuclear aromatic hydrocarbons(PAHs) in air particulate matter [CHD 32: Environmental Protection and Waste Management]



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भारतीय मानक

वायु प्रदूषण मापने की पद्धति

भाग 12 वायु विषक्त तत्वों में बहुपरमाणु एरोमैटिक हाइड्रोकार्बन (पीएएचएस)

(पहला पुनरीक्षण)

Indian Standard

METHOD FOR MEASUREMENT OF AIR POLLUTION

PART 12 POLYNUCLEAR AROMATIC HYDROCARBONS (PAHs) IN AIR PARTICULATE MATTER

(First Revision)

ICS 13.040.20

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

November 2004 **Price Group 4**

FOREWORD

This Indian Standard (Part 12) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Environment Protection and Waste Management Sectional Committee had been approved by the Chemical Division Council.

Polycyclic aromatic hydrocarbons (PAHs) an ubiquitous environmental pollutant that represent the largest class of suspected chemical carcinogens, PAHs are compound of two or more benzene rings with adjacent rings sharing two carbon atoms, non-aromatic rings may also be present. Physico-chemical characteristics of PAHs are highly governed by their molecular weight. Resistance to oxidation and reduction, varpour pressure and aqueous solubility, for instance, decrease with increasing molecular weight.

The PAHs can be formed from both natural and anthropognic sources. With the exception of anthracene, which was used in the dye industry, PAHs have no commercial use except for research purposes. Apart from small amounts of geothermal and biosynthetic origin (synthesized by some algae, bacteria, fungi and plants), PAHs are mainly anthropogenic. Heating, power plants using fossil fuels, industrial process, incineration of industrial and domestic wastes, forest fires, volcanic activity, vehicle exhausts, asphalt pavements, and in general, all incomplete combination at high temperature and pyrolytic processes involving fossils fuels or more generally, materials containing carbon and hydrogen, are major sources of PAHs.

Human exposure to PAHs occurs principally by inhalation of tobacco smoke and polluted air, ingestion of contaminated and processed food and water, or by dermal contact with soots, tars and oils. The greatest exposure is likely to take place in the workplace of certain facilities like tar production plants, coking plants, asphalt production plants, coal-gasification sites, smoke-houses, municipal trash incinerators via air. In air, PAHs may exist adbsorbed on carrier particles or in free state.

This standard was first published in 1974 based on ASTM D 2682 : 1972 'Method of test for polynuclear aromatic hydrocarbons in air particulate matter'. This revision has been necessitated in view of the latest developments which have taken place in the sampling techniques and the need to have results for ambient air quality expressed as nanogram per cubic metre and for fugitive and source emissions results expressed as micro-gram per cubic metre. Gas chromatographic (GC) method using flame ionization detector (FID) is provided in this standard. Other analytical methods like HPLC and GC-MS may be considered by the committee for formulation later on.

Considerable assistance has been derived from ISO/DIS 12884 : 1997 'Air quality Determination of gas and particle-phase polycyclic aromatic hydrocarbons in ambient air collection on sorbent-backed filters with gas chromatographic/mass spectrometric analysis'.

The composition of the Committee responsible for formulation of this standard is given at Annex B.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHOD FOR MEASUREMENT OF AIR POLLUTION

PART 12 POLYNUCLEAR AROMATIC HYDROCARBONS (PAHs) IN AIR PARTICULATE MATTER

(First Revision)

1 SCOPE

1.1 This standard (Part 12) describes a method for sampling and analysis procedure for PAHs in ambient air and fugitive emission.

1.2 It does not measure vapour-phase PAH, for that polyurethane foam plugs may be used after the filter for sampling.

2 PRINCIPLE

This method is designed to collect particulate phase PAHs in ambient air and fugitive emissions and to determine individual PAH compounds. It is based on high volume (~ 1.2 m^3/min) sampling method capable of detecting sub ng/m^3 concentration of PAH with a total sample volume ~ 480 m^3/ of air over a period of 8 h with same filter. It involves collection from air particulate on a fine particle (glass-fibre) filter using high volume sampler for total suspended particulate matter (TSPM) or respirable dust sampler for respirable suspended particulate matter (RSPM or PM10) and subsequent analysis by Gas Chromatograph (GC) using Flame Ionization Detector (FID). If sampling period is extended to 24 h without changing the filter, it may enhance sample loss due to volatility or reactions of PAHs on collection media.

3 INTERFERENCES

3.1 The particle phase PAH may be lost from particle filter during sampling due to desorption and volatilization especially during summer months at ambient temperature of 30°C and above.

3.2 The method interference may be caused by contaminations in low grade filter, solvent, and reagent, if used.

3.3 Glassware shall be properly cleaned (acid-washed) followed by solvent rinsing prior to use.

3.4 Matrix interferences may be caused by contaminants, that is, hydrocarbons and other organics that are co-extracted from sample. In this case clean-up by column chromatography shall be required besides identification and confirmation of individual analyte followed by mass-spectrometer.

4 SAMPLE PRESERVATION

Sample should be wrapped in an aluminium-foil and should be stored in a refrigerator at 4°C in dark place to avoid photo-oxidation of PAHs for a period up to two months. However, sample extracts may be strored in dried form for a longer period.

5 APPARATUS

5.1 Ultrasonicator, with compact tank/bath of 4.5 litre capacity and producing ~ 40 kHz frequency for extraction.

5.2 Rotary Evaporator, buchi-type.

5.3 Silica-Gel Column, 200 mm length, 5 mm internal diameter with teflon stopcock.

5.4 GC-FID with Capillary Column

5.5 Syringes, 1 µl to 10 µl.

5.6 Flask and Beakers, 5-ml, 10-ml, 25-ml, 50-ml and 250-ml capacity.

5.7 Variable Volume Micro-Pipettes, 0.5 ml and 1.0 ml capacity.

6 REAGENTS

6.1 All solvents to be used should be of reagent grade.

6.1.1 *Toluene*, ultra-residue grade.

6.1.2 *Cyclohexane*, ultra-residue grade.

6.1.3 *Tri-phenyl Benzene*, ultra-residue grade.

6.1.4 *Solid PAHs Compounds*, high purity (*see* Annex A) to prepare the standard PAH solution.

6.1.5 *Activated Silica Gel* (60-100 meshes), chromatography grade.

7 PROCEDURE

7.1 Sampling

Collect sample through a high volume-sampler (HVS) using glass fibre (EPM — 2000) filter paper perferably Whatman or equivalent) at a flow rate of ~ 1.2 m^3/min over an extended period of time usually 8 h for ambient air.

NOTE — A multi-point calibration of flow control system shall be conducted every 3 months (maximum 6 months) using standard calibration orifice/device.

7.2 Sample Processing and Extraction

Cut/punched at least 30 percent of total sample of the exposed filter paper or measured fraction of it into small strips/circular pieces in a beaker/flask of 250-ml capacity. Add tri-phenyl benzene, an internal standard at this stage for recovery test. Add about 100 ml of toluene for extraction and keep beakers in ultrasonic bath for 30 min (or for 6 using Soxhlet extraction apparatus). Filter the extracts into evaporative flask of 250 ml with the help of Whatman filter paper No. 20 or filter-disc. Repeat the extraction twice and combine extractants.

7.3 Sample Concentration

Evaporate the toluene extracts using rotary evaporator with water bath as cool as possible (temperature not exceeding 40°C). Do not evaporate up to total dryness. It should be stopped at near dryness (less than 1 ml, visible). Add 2.0 ml of toluene to rinse the wall of evaporation flask and transfer extract into a beaker of 5 ml capacity.

NOTE — Samples extraction should preferably be carried out within a month of sampling.

7.4 Clean-Up and Enrichment

It is performed using silica gel column having length 200 mm, and inner diameter (ID) 0.5 cm. Pour a slurry of 3 g deactivated silica gel (60-100 mesh size) in cyclohexane into the column. Elute toluene followed by cyclohexane through the column for conditioning. Now introduce sample extract (concentrated, 2.0 or 3.0 ml) at the top of silica column. Collect the PAH fraction with about 5 ml of cyclohexane. Collect all the eluants into a rotary evaporator flask. Add another 30 ml of cyclohexane to the column to elute all organics of interest. Collect all fractions into the flask and reduce to about 1 ml. Finally transfer into 5 ml capacity beaker/vials, dry and store in a dark and cool place.

7.5 Gas Chromatographic Analysis

Dilute the extracted residue and make up to 0.5 ml or 1 ml. Inject 1 µl or 2 µl into GC-FID for analysis.

7.6 Gas Chromatography Conditions

7.6.1 Gas chromatograph equipped with percent ionization detector (FID), a split injector and capillary column (Phase cross linked 5 percent phenyl, methyl-silicone) : 25 m length, 0.2 mm inner diameter (ID), 0.33 µm film thickness with following GC conditions:

Injection — Port — Temperature : 320°C
FID — Temperature : 320°C

Oven — Temperature — Programme : Initial temperature 140°C, hold for 3 min with :

	Rate Deg/min	*Final Temperature* °C	*Hold for* min
Ramp A	6	250	6
Ramp B	10	300	5

Total run time : 36 min

NOTE — Temperature programming may vary to suit better resolution of peaks and subsequently also the total run time based upon column features.

7.6.2 *Carrier Gas*

Nitrogen linear flow 30 ml/min (column flow 1 : 2 ml/min, make up flow 28.8 ml/min), split ratio 1 : 20 approximately.

7.6.3 *Gases for FID*

Hydrogen flow 30 ml/min and air flow approximately 300 ml/min

7.6.4 *Type of Liner*

Glass liner with some glass wool.

7.6.5 *Integrator Setting*

Chart speed : 0.5 cm/min; Attenuation : Zero; Peak Window : Zero; Range : 8; Thresh : Zero; Area Rejection : 50; Baseline : 10 Ref RT window : -0.08; Non-Ref RT window : -0.08

7.6.6 *Calibration of GC*

7.6.6.1 *Stock standard solution*

Use solid PAH standards (10 mg/10 ml of toluene) to obtain the stock solution of PAH.

7.6.6.2 *Working standard solution*

From stock solution, make a working standard solution of PAH by taking 625 µl of each individual PAH compound into a 25-ml volumetric flask and make up with toluene (concentration should be 25 µg/ml). This PAH working solution is used for calibration of GC.

7.6.6.3 *Internal calibration*

Each of the calibration standards shall contain the appropriate internal standards as specified concentration. Analyze (1 or 2 µl) of each standard

solution and plot the area ratio of analyte and the corresponding internal standard against the concentration for each compound and internal standard.

7.6.6.4 The response factor (RF) for each analyte is calculated using the following equation:

$$RF = (A_s \times C_{is}) / (A_{is} \times C_s)$$

where

A_s = area of the analyte (sample/standard) to be measured,

A_{is} = area of the internal standard,

C_{is} = concentration of the internal standard (ng/µl), and

C_s = concentration of the analyte (sample/ standard) to be measured (ng/µl).

7.6.6.5 Add internal standards to all calibration standards and sample extracts analysed by GC. If the RF is constant over the working range (< 20 percent RSD), assume the RF to be invariant and use the average RF for calculations. Alternatively, plot the results a calibration curve of response ratios, that is A_s/A_{is} vs. *RF*.

7.6.6.6 Verify the working calibration curve or RF on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ± 20 percent, repeat the test using a fresh calibration standard. Alternatively, prepare a new calibration curve. The relative retention times for each compound in each calibration run shall agree within 0.03 relative retention time units.

7.6.6.7 When the ratio (*R*) of the retention time (*RT*) of the unknown analyte, T_u to that of the corresponding internal standard (where $R = Rt_u / Rt_i$) is used to identify the analyte, the ratio of retention times (R_S) from the sample chromatogram shall not be greater than 0.4 percent of the retention time ratio (R_C) from the chromatogram of the calibration standard. The value of *R* shall not be more than 2 or less than 0.5. The retention index of the sample analyte and corresponding standard shall agree within ±2 percent.

7.6.6.8 *External calibration*

When an analyte has been identified, the quantification of that analyte will be based on peak area response of respective compounds with respect to working calibration standard, that is, calibration factor (the ratio of response to the amount of mass injected). The retention times of various PAHs compounds obtained under the above GC condition are given in Fig. 1.

7.6.6.9 *Sample analysis*

Dilute the extracted residue and make up to 0.5 ml or 1 ml. Inject 1 µl or 2 µl into GC- FID for analysis and record the resulting peak size in area units / (or peak height when overlapping peaks cause errors in area integration). Identify the compounds using external calibration procedures from the sample chromatograms.

NOTES

1 Confirmation may be required on second GC column or by other technique, that is GC-MS if overlaps of peaks occur.

2 Validation of GC system is performed through the use of mid range standards. If it falls outside its daily retention time window, the system is out of control. The cause of problem is detected and error is rectified. A new calibration is to be performed whenever a system is adjusted.

7.6.6.10 *Field blank*

Use atleast 10 percent of the samples or minimum of one per batch of samples as field blank.

8 CALCULATION

8.1 Calculate the concentration in (ng/µl) of each identified analyte in the sample extract (C_s) as follows:

$$C_s = (A_s \times C_{is}) / (A_{is} \times R_F)$$

where

A_s = area count of characteristic analyte sample/peak being measured,

A_{is} = area count of characteristic internal standard/peak, and

C_{is} = concentration of internal standard.

8.2 Calculate the air volume from the periodic flow reading taken during sampling using the following equation:

V = Average flow rate of sampling, $m^3/min \times T$

where

V = total sample volume at ambient conditions, in m^3; and

T = elapsed sampling time, in min.

8.3 The volume of air sampled (V_s) may optionally be converted to standard conditions of temperature and pressure (25°C and 101 kPa) using the following equation:

$$V_s = V \times (P_a / 101) \times [298/(273 + T_a)]$$

where

V = total sample volume under ambient conditions, in m^3;

P_a = ambient pressure, in kPa; and

T_a = ambient temperature, in °C.

8.4 The concentration, in ng/m^3, of each analyte in the air sampled is given by:

$$C = C_s \times V_e / Vs$$

where

V_e = final volume of extract, in µl; and

V_s = volume of air sampled (*see* **8.3**).

9 DETECTION LIMIT

The minimum detectable concentration in term of BaP for a sampling period of 8 h (with about 480 m^3 of air passed) will be 2 ng per cubic meter assuming 0.5 ml as the final volume of sample extract after clean-up and detectable concentration of 2 ng/µl of that sample extract. High resolution mass-spectrometry or high pressure liquid chromatography can improve sensitivity down to 1ng/m^3.

10 DETERMINATION OF SAMPLING EFFICIENCY (SE)

10.1 Confirm the efficiency of the sampler for the targeted PAH under the conditions anticipated in the field prior to the initiation of any sampling programme. Determination of the efficiency is particularly important if sampling periods exceeding 24 are planned. Acceptable performance may be established by determining sampling efficiency.

10.2 Determine sampling efficiency by spiking a solution of the compounds of interest (or a representative selection that includes the most volatile PAH) onto a clean filter, then pulling through the assembled sampling module a volume of air equivalent to the maximum volume that will be sampled.

10.3 Add the spiking solution for SE determination dropwise to the filter, so as to uniformly load it and avoid oversaturation. Spiking levels shall correspond to atleast 3 times but not more than 10 times at anticipated concentrations of the targeted compounds in the air to be pulled through the sampling medium. Allow the spiked filter or sorbent to dry for about one hour in a clean, light-protected area prior to pulling air through the system.

10.4 The sampling rate and sampling period shall be the same as that planned for the programme. Ambient temperatures during the test shall also approximate those expected in the field, especially when warm-weather conditions are anticipated.

11 QUALITY ASSURANCE TEST

11.1 Recovery efficiencies (*RE*) of the isotopically-labelled PAH surrogates added to the samples prior to extraction and analysis shall be closely monitored to assure the effectiveness of sample work-up and analytical procedures. The surrogate recoveries should fall between 75-125 percent. Discard the samples for which surrogate recoveries are less than 50 percent or more than 150 percent.

11.2 Approximately 10 percent of the sample extracts shall be subjected to duplicate GC analysis to assure acceptable analytical precision.

12 EXPRESSION OF RESULTS

Express results for ambient air quality as nanogram per cubic metre (ng/m^3) of air whereas for fugitive and source emissions express results in micro-gram per cubic metre of air.

13 PRECISION

Precision and uncertainty under normal condition can be expected to be ±30 to ±45 percent. Precision will vary with sample volume and analyte concentration. An overall mean standard deviation of about ±15 to ±20 percent is acceptable air samples over a period for different PAH concentration range of up to 50 ng/m^3.

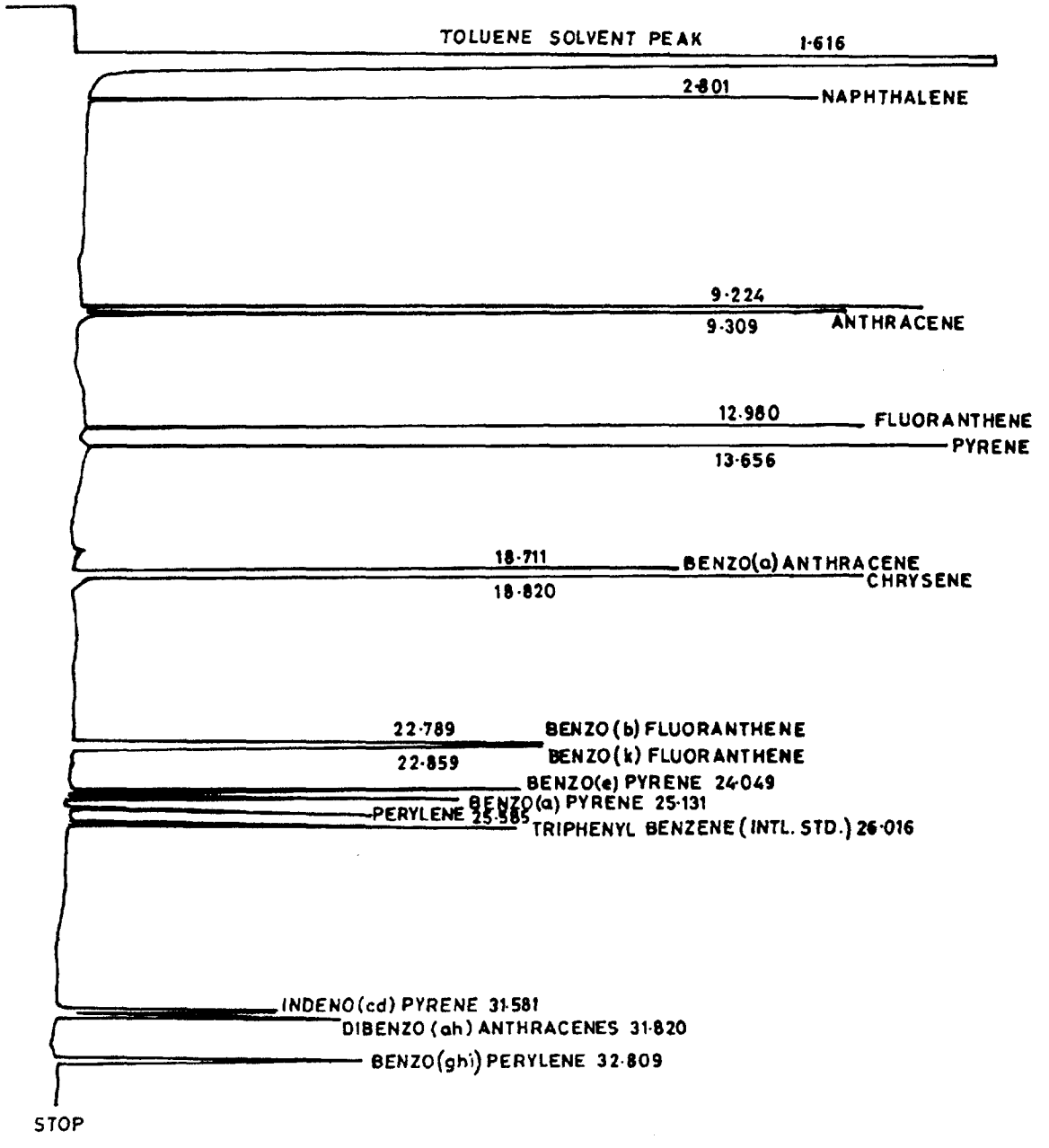

FIG. 1 CHROMATOGRAM OF STANDARD PAH MIXTURE

ANNEX A

(*Clause* 6.1.4)

PURITY OF POLYCYCLIC AROMATIC HYDROCARBONS (SOLID COMPOUNDS FOR MAKING CALIBRATION STANDARDS)

Sl No.	*Polycyclic Aromatic Hydrocarbons (PAHs)*	*Molecular Formula*	*Purity/Grade*	*Molecular Weight*
(1)	(2)	(3)	(4)	(5)
i)	Naphthalene	$C_{10}H_8$	99+ %	128
ii)	Phenanthrene	$C_{14}H_{10}$	99+ %	178
iii)	Anthracene	$C_{14}H_{10}$	99+ %	178
iv)	Fluoranthene	$C_{16}H_{10}$	98+ %	202
v)	Pyrene	$C_{16}H_{10}$	98+ %	202
vi)	Chrysene	$C_{18}H_{12}$	95+ %	228
vii)	Benzo (a) anthracene	$C_{18}H_{12}$	99+ %	228
viii)	Benzo (b) fluoranthene	$C_{20}H_{12}$	99+ %	252
ix)	Benzo (k) fluoranthene	$C_{20}H_{12}$	98+ %	252
x)	Benzo (e) pyrene	$C_{20}H_{12}$	99+ %	252
xi)	Benzo (a) pyrene	$C_{20}H_{12}$	98+ %	252
xii)	Perylene	$C_{20}H_{12}$	98+ %	252
xiii)	Benzo (ghi) perylene	$C_{22}H_{12}$	98+ %	276
xiv)	Dibenzo (ah) anthracenes	$C_{22}H_{14}$	98+ %	278
xv)	Indeno (cd) pyrene	$C_{22}H_{12}$	99+ %	276
xvi)	Coronene	$C_{24}H_{12}$	98+ %	300

NOTE — Alternatively readymade Reference PAH Standard Mixture in liquid form available in ampoules with traceable purity may be obtained.

ANNEX B

(Foreword)

COMMITTEE COMPOSITION

Environment Protection and Waste Management Sectional Committee, CHD 32

Organization	*Representative(s)*
In personal capacity (*40/14, C. R. Park, New Delhi 110019*)	PROF DILIP BISWAS (***Chairman***)
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