

USEPA  
Hazardous Waste Support Branch  
Validating Air Samples  
Volatile Organic Analysis Of Ambient Air In Canister  
By Method TO-15



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Annual Review

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S))  
YES NO N/A

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: \_\_\_\_\_ SDG(s): \_\_\_\_\_  
 SITE: \_\_\_\_\_ LAB: \_\_\_\_\_

This Region II SOP document is based on Method TO-15: Determination of Volatile Organics Compounds (VOCs) in Air Collected in Specially-Prepared Canisters & Analyzed by Gas Chromatography/Mass Spectrometry, January 1999.

**1.0 Data Completeness and Deliverables**

1.1 Have any missing deliverables been received and added to the data package? [ ] \_\_\_ \_\_\_

ACTION: Contact lab for explanation/resubmittal of any missing deliverables. If lab cannot provide them, note the effect under "Contract Problems/Non-Compliance" section of data assessment report.

**2.0 Cover Letter, Narrative, and Data Reporting Forms**

2.1 Is the Lab. Narrative and Cover Page present?	[ ]	___	___
2.2 Is Case Number contained in the Narrative?	[ ]	___	___
2.3 Are the following Data Reporting Forms present?			
Analysis Data Sheet [Form I/Equivalent]	[ ]	___	___
Tentatively Identified Compounds [Form I-TIC]	[ ]	___	___
Blank Summary [Form IV/Equivalent]	[ ]	___	___
Laboratory Control Sample Data Sheet [Form III/Equivalent]	[ ]	___	___
GC/MS Instrument Performance Check and Mass Calibration [Form V/Equivalent]	[ ]	___	___
Initial Calibration [Form VI/Equivalent]	[ ]	___	___
Continuing Calibration [Form VII/Equivalent]	[ ]	___	___
Internal Standard Area and RT Summary [Form VIII/Equivalent]	[ ]	___	___

YES NO N/A

Canister Certification [Form IX/Equivalent] [ ] \_\_\_ \_\_\_

**3.0 Canister Receipt/Log-in Sheet**

Receipt of each canister is recorded in a laboratory notebook dedicated to this use. The sample receipt/log-in sheet must demonstrate that the information on custody records, traffic reports, and sample tags agree for each sample.

3.1 Do all info items agree with each sample ? [ ] \_\_\_ \_\_\_

ACTION: If these documents are not consistent, contact Project officer or laboratory and attach a record of resolution.

**4.0 Traffic Reports and Laboratory Narrative**

4.1 Are the Traffic Report Forms present for all samples? [ ] \_\_\_ \_\_\_

ACTION: If no, contact lab for replacement of missing or illegible copies.

**5.0 Holding Times**

5.1 Have any VOA technical holding times of 30 days, determined from the date of sample collection to the date of analysis, been exceeded? \_\_\_ [ ] \_\_\_

NOTE: The contract requires that samples must be retained from verified time sample receipt (VTSR) until 45 days after delivery of a complete sample data package to the Agency.

VOA Table of Holding Time Violations

Sample ID	Sample Matrix	Date Lab Received	Date Analyzed
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

YES NO N/A

ACTION: If technical holding times have been exceeded,  
flag all results unusable ("R").

**6.0 Leak Test Evaluation**

6.1 All canisters are leak tested prior to each  
sampling use.  
Form IX/Equivalent - summarizes the canister  
certification for each canister. The initial  
gauge pressure should be approximately 206 kPa  
(30 psi) with zero air.

Did the pressure test not vary by more than  
 $\pm 13.8$  kPa ( $\pm 2$  psi) over the 24 hours period? \_\_\_\_\_  \_\_\_\_\_

ACTION: If the canister does not meet the leak-tight  
criteria all results should be flagged "R".

**7.0 Canister Certification Form IX/Equivalent**

7.1 Blank Analysis

All canisters have to be checked after cleaning.

Were the target analytes < the required detection  
limits specified in the task order?  \_\_\_\_\_

Note: Samples with large amount of non target  
analytes can be valid as long as this  
criterion is met for target analytes.

ACTION: If the lab failed to do so, it should be noted  
under contract non-compliance, and laboratory  
should be notified. Use Table 1 below to qualify  
samples with target compounds results also present  
in certification blanks.

**Certification Contamination  
 TABLE 1**

Certification Contamination	Sample Result	Action for Sample
≥ detect limit specified in task order	> 5X certification contamination	No qualification required
≥ detect limit specified in task order	< detect limit specified in task order	detection limit with U
≥ detect limit specified in task order	≥ detect limit and ≤ 5X certification contamination level	5X certification contamination with U
< detect limit specified in task order	≤ detection limit and ≥ detection limit	no qualification

7.2 Is the canister certification form provided, and the associated canister sample identification included?  
 When contamination, included contamination detected  
 (all raw data),analyte and reference mass spectra. [ ] \_\_\_ \_\_\_

ACTION: If no, have EPA project officer/TOPO contact laboratory for missing documents.

**8.0 Laboratory Control Samples**

8.1 Is an LCS Data Sheet (Form III/Equivalent) present and complete for each LCS? [ ] \_\_\_ \_\_\_

8.2 Was an LCS prepared (10ppbv total scan) (0.1ppbv SIM) and analyzed at the required frequency (once per 24 hour analytical sequence, and concurrently with the samples in the SDG)? [ ] \_\_\_ \_\_\_

ACTION: Call lab for explanation/resubmittals.  
 If missing deliverables or information is unavailable, document the effect in the data assessment.

8.3 Are there any transcription/calculation errors between the raw data and Form III/Equivalent?

YES NO N/A

Check LCS target compound recoveries.

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document the effects in the data assessment.

8.4 Is the % recovery within 70-130 % for each LCS target compound reported on Form III/Equivalent?

ACTION: Professional judgement should be used to qualify the impact on sample data, if the recoveries are outside the given limits.

8.5 Is the RT of each reported LCS compound within the windows established during the most recent valid calibration?

If the most recent calibration is the initial calibration use mid level standard (10 ppbv).

ACTION: Professional judgement should be used to qualify sample data, if retention times differ by more than 20 seconds.

8.6 Do the Internal Standards meet the requirements specified in Sections 18.1 and 18.2?

ACTION: If not, see Sections 18.1 and 18.2.

ACTION: Circle outliers in red.

ACTION: Always use professional judgement. If qualification is necessary, follow the criteria below and in Table 2.

1. If any LCS compounds are outside the specified limits, the associated sample results for the outlying compounds should be qualified as indicated in Table 2 below.
2. If the absolute RT for any LCS compound is outside the established windows, then qualify positive results and non-detects in the associated environmental sample data for that LCS compound(s) (See Table 2). All non-LCS compounds should be qualified using professional judgement.

**Laboratory Control Samples  
 TABLE 2**

The following table summarizes the LCS criteria and the data qualification guidelines for all associated field samples.

LCS	<u>NOT QUALIFIED</u>	<u>J</u>	<u>R</u>
% RECOVERY			
Detects	70 - 130%	< 70%, > 130%	
Non-detects	≥ 130%	50 - 69%	< 50%
ABSOLUTE RT OF LCS COMPOUNDS			
LCS Compounds in samples RT: (min)	± 0.33		> ± 0.33

**9.0 GC/MS Instrument Performance Check**

9.1 Are the GC/MS Instrument Performance Check

Forms (Form V/Equivalent) present for  
 Bromofluorobenzene (BFB)?  \_\_\_\_

9.2 Are the enhanced bar graph spectrum and  
 mass/charge (m/z) listing for the 50 ng BFB  
 provided for each twenty four hour shift?  \_\_\_\_

9.3 Has the instrument performance compound been  
 analyzed for every twenty four hours of sample  
 analysis per instrument?  \_\_\_\_

ACTION: List date, time, instrument ID, and sample analysis for which no associated GC/MS tuning data are available.

YES NO N/A

DATE	TIME	INSTRUMENT	SAMPLE NUMBERS
_____	_____	_____	_____
_____	_____	_____	_____

ACTION: If lab cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

9.4 Have the ion abundances been normalized to m/z 95?  \_\_\_ \_\_\_

ACTION: If mass assignment is in error, qualify all associated data as unusable (R).

9.5 Have the ion abundance criteria been met for each instrument used?  \_\_\_ \_\_\_

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If ion abundance criteria are not met, the Region II TPO must be notified.

9.6 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.) \_\_\_  \_\_\_

9.7 Have the appropriate number of significant figures (two) been reported?  \_\_\_ \_\_\_

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.

9.8 Are the spectra of the mass calibration compound acceptable?  \_\_\_ \_\_\_

ACTION: Use professional judgement to determine whether associated data should be accepted, or qualified.

**10.0 Performance Evaluation Sample (Optional)**

10.1 The PE sample will assist the Agency in monitoring Contractor performance. The lab will not be informed as to which compounds are contained in the

YES NO N/A

PE samples or the concentrations. Was a PE sample submitted from the Agency with each SDG?

10.2 PE samples must be validated like environmental samples. There is no holding time for PE samples. If the data results do not comply with the Agencies' spike results use professional judgement together with other QC criteria in order to determine usability of the other data in the SDG. If the associated data was rejected because of PE results, the EPA technical project officer must be notified.

10.3 Do the Internal Standards meet the requirements specified in Sections 18.1 and 18.2?

ACTION: If not, see Sections 18.1 and 18.2.

**11.0 Laboratory Method Blanks**

11.1 Is an Analysis Data Sheet (Form IV/Equivalent) present and complete for each method blank?

11.2 Frequency of analysis:  
 Has a method blank analysis been reported per instrument for each 24-hour analytical sequence?     
 Has a method blank been analyzed after the initial calibration or a valid calibratio check standard, and before the LCS, prior to sample analysis?

ACTION: If any blank data are missing, call lab for explanation/resubmittals. If missing deliverables are unavailable, reject ("R") all positive data.

11.3 Chromatography: review the blank raw data - chromatograms, quant reports and data system printouts. Is the chromatographic performance (baseline stability) for each instrument acceptable?

ACTION: Use professional judgement to determine the effect on the data.

11.4 Were the area response of each Internal Standards (IS) in the blank within  $\pm 40\%$  of the mean area response of the IS of the most recent valid calibration?     
 Were the RT of each IS within  $\pm 0.33$  min (20 sec.) between blanks & most recent valid calibration

ACTION: If not, see section 18.1 and 18.2.

**12.0 Blank Contamination**

12.1 Do any method blanks have positive target and non-target VOA results ? \_\_\_  \_\_\_

ACTION: Use Table 3 below to qualify samples with target compound results also present in the associated blank. Use the largest value from all the associated method blanks if more than one method blank was run.

**VOA Laboratory Blanks  
TABLE 3**

Samples	Not Qualified	non detect U
Target Compounds	> 5X Blank value	≤ 5X Blank Level*

\* If sample result is also less than CRQL, report as not detected (U) at [CRQL]. Note that the dilution factor has to be taken into account when calculating the Blank Level.

**13.0 Target Compound Analytes**

13.1 Are the Organic Analysis Data Sheets (Form I-, Equivalent), VOA chromatograms, and data system printouts present and complete with required header information for each of the following:

a. Samples?	<input type="checkbox"/>	___	___
b. Method blanks?	<input type="checkbox"/>	___	___
c. Laboratory Control Sample (LCS)?	<input type="checkbox"/>	___	___
d. Performance Evaluation Sample (PES)?	<input type="checkbox"/>	___	___

ACTION: If any data are missing, take action specified in 1.1 above.

13.2 Is chromatographic performance acceptable with respect to:

a. Baseline stability?	<input type="checkbox"/>	___	___
b. Resolution?	<input type="checkbox"/>	___	___
c. Peak shape?	<input type="checkbox"/>	___	___
d. Full-scale graph (attenuation)?	<input type="checkbox"/>	___	___
e. Other:	<input type="checkbox"/>	___	___

13.3 Were any electropositive displacement (negative peaks) or unusual peaks seen? \_\_\_  \_\_\_

YES NO N/A

ACTION: Use professional judgement to determine the acceptability of the data. Address comments under "System Performance" section of data assessment.

13.4 Is the sample component relative retention time (RRT) within  $\pm 0.06$  RRT units of the RRT of the standard component from the most recent continuing calibration?

NOTE: If the most recent calibration is a calibration curve, the mean RRT (RRT) should be used for comparison.

ACTION: If the above criteria is not met, professional judgement should be used to qualify sample data.

13.5 Was Nafion dryer used?

ACTION: In cases where Nafion tubing is used to dry the sample stream, polar target and non target compounds must not be reported.

ACTION: Reject all polar compounds if reported as non detects. Polar compounds reported as positive hits should be flagged "J".

**14.0 Tentatively Identified Compounds (TIC)**

14.1 Are all Tentatively Identified Compound Forms (Form I-TIC) present and are retention time, estimated concentration and "JN" qualifier listed corresponding to each TIC?

14.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following?

a. Samples

b. Blanks

ACTION: If any TIC data are missing, take action specified in 1.1 above.

ACTION: Add "JN" qualifier if missing.

14.3 Are all ions present in the reference mass spectrum with a relative intensity greater

YES NO N/A

than 10% also present in the sample mass spectrum?

14.4 Do TIC and "best match" standard relative ion intensities agree within 20%?

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate.

Also, when a compound is not found in any blanks, but is detected in a sample and is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable (R). (e.g., Common Lab Contaminants: CO<sub>2</sub> (M/E 44), Siloxanes (M/E 73), Aldol Condensation Products, Solvent Preservatives, and related by products.

**15.0 Initial Calibration and System Performance (Form VI/Equivalent)**

15.1 Were each GC/MS system calibrated at 5 concentrations that span the monitoring range of interest in an initial calibration sequence to determine the sensitivity and the linearity of the GC/MS response for the target compounds?

ACTION: If any calibration standard forms or raw data are missing, take action specified in section 1.1 above.

15.2 Was the same volume introduced into the trap consistently for all field and QC-sample analyses?

15.3 Were the area response (Y) at each calibration level within  $\pm 40\%$  of the mean area response (mean Y) over the initial calibration range for each Internal Standard?

Did the laboratory tabulate the area response (Y) of the primary ions and the corresponding concentration for each compound and Internal Standard?

ACTION: If the range exceeds  $\pm 40\%$  for particular compounds, flag these compounds "J" for

positive and non-detects in the associated samples. If the %RSDs exceeds  $\pm 90\%$ , associated sample non-detect compounds should be rejected (R) and associated hits as estimate (J).

15.4 Are the relative retention times (RRT) for each of the target compounds at each calibration level within  $\pm 0.06$  RRT units of the mean relative retention time for the compound?

ACTION: If no, reject the associated sample compounds.

15.5 Are all individual RRF and average RRFs  $\geq 0.050$ ?

NOTE: For the following compounds the individual RRF and average RRF must be  $\geq 0.01$ .

- 2-Butanone
- Carbon disulfide
- Chlorethane
- Chlormethane
- 1,2-Dibromoethane
- 1,2-Dichloropropane
- 1,4-Dioxane
- 1,2-Dibromo-3-chloropropane
- Methylene chloride

ACTION: Circle all outliers with red pencil.

ACTION: For any target analyte with average RRF  $< 0.05$ , or for the requirements for the 9 compounds in 15.5 above, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

15.6 Are response factors (RF) stable i.e. % Relative Standard Deviation (%RSD)  $\leq 30.0\%$  with at most two exceptions up to limit of  $\pm 40\%$ ?

ACTION: Circle all outliers in red.

ACTION: If %RSD  $> 30.0\%$ , qualify associated positive results for that analytes "J" and non-detects are not qualified. When RSD  $> 90\%$ , flag all non-detects for that analytes R (unusable) and associate positive values as estimate (J).

NOTE: Analytes previously qualified "U" for blank contamination are still considered

YES NO N/A

as "hits" when qualifying for initial calibration criteria.

15.7 Are there any transcription/calculation errors in the reporting of average response factors (RRFs) or %RSDs? (Check at least 2 values, but if errors are found, check more.) \_\_\_ [ ] \_\_\_

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effects in data assessment.

15.8 Are the RT shift for each Internal Standard (IS) at each calibration level within 20s of the mean RT over the initial calibration range of each IS? [ ] \_\_\_ \_\_\_

**16.0 Daily Calibration (Form VII/Equivalent)**

16.1 Are the daily Calibration Forms (Form VII/Equivalent) present and complete for the volatile fraction? [ ] \_\_\_ \_\_\_

16.2 Has a daily calibration standard (10 ppbv total scan) (0.1ppb SIM) been analyzed for every twenty four hours of sample analysis per instrument after the BFB tuning analysis? [ ] \_\_\_ \_\_\_

ACTION: List below all sample analyses that were not within 24 hours of the daily calibration analysis.

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

ACTION: If any forms are missing or no daily calibration standard has been analyzed within 24 hours of every sample analysis, call lab for explanation/resubmittal. If daily calibration data are not available, flag all associated sample data as unuable ("R").

16.3 Do any volatile compounds have a % Difference (% D) between the initial and daily RRFs which exceed the  $\pm 30\%$  criteria? \_\_\_ [ ] \_\_\_

YES NO N/A

ACTION: Circle all outliers in red.

ACTION: Qualify both positive results and non-detects  
for the outlier compound(s) as estimated (J).  
When % D is above 90%, reject non-detects as R)  
unusable and associated positive values (J).

16.5 Are there any transcription/calculation  
errors in the reporting of average response  
factors (RRF) or %difference (%D) between  
initial and daily RRFs? (Check at least  
two values but if errors are found,  
check more.) \_\_\_ [ ] \_\_\_

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for  
explanation/resubmittal, make any  
necessary corrections and note errors  
under "Contract Non-Compliance".

**17.0 Compound Quantitation and Reported Detection Limits**

17.1 Are there any transcription/calculation errors in  
Form I results? Check at least two positive values.  
Verify that the correct average RRF of the initial  
calibration was used to calculate Form I results. [ ] \_\_\_ \_\_\_

17.2 Are the reported detection limits adjusted to  
reflect sample dilutions? [ ] \_\_\_ \_\_\_

ACTION: If errors are large, call lab for  
explanation/resubmittal, make any necessary  
corrections and note errors under "Contract  
Non-Compliance" of the data assessment.

NOTE: When a sample is analyzed at more than  
one dilution, the lowest CRQLs are used  
(unless a QC accedence dictates the use  
of the higher CRQL data from the diluted  
sample analysis). Cross out "E" from the  
original analysis. Replace the concentrations  
in the original analysis with the ones from  
the diluted sample. Specify which Form I  
is to be used. Draw a red "X" across the entire  
page of all Form I's that should not be used,  
including any in the summary package.

17.3 Have any target compound concentrations exceeded  
the calibration range of the GC? \_\_\_ [ ] \_\_\_

ACTION: If yes, flag as estimated ("J").

- 17.4 Was more than one method of quantitation used to calculate sample results within a batch or 24 hr. analytical sequence? \_\_\_ [ ] \_\_\_
- 17.5 Did the lab report the target compounds below CROLs with the suffix "J"? [ ] \_\_\_ \_\_\_

ACTION: When appropriate, include suffix "J".

**18.0 Internal Standard (Form VIII/Equivalent)**

- 18.1 Are the 3 internal standard areas (Form VIII) of every sample, LCS, PE, and blank within the upper and lower limits (+40% to -40%) for each continuing calibration or 10 ppbv level of initial calibration? [ ] \_\_\_ \_\_\_

ACTION: List all the outliers below.

Sample #	Internal Std	Area	Lower Limit	Upper Limit
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

- ACTION:
1. If the internal standard area count is outside the limit, flag all positive results quantitated with this internal standard with a "J."
  2. Non-detects associated with IS area counts > 40% are not qualified.
  3. If IS area is below the lower limit (< 40%), qualify all associated non-detects (U values) "J". If extremely low area counts are reported, (< 25%), or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable ("R").

- 18.2 Are the internal standard retention times in each sample, LCS, PE, and blank within 20 seconds of the corresponding retention times in the associated calibration standard? [ ] \_\_\_ \_\_\_

ACTION: Professional judgement should be used to qualify sample data if the internal standard

retention times differ by more than 20 seconds.

**19.0 Mass Spectral Interpretation/Identification**

19.1 Are the Organic Analysis Data Sheets present with required header information on each page, for each of the following:

- a. Samples and/or fractions as appropriate?
- b. Laboratory Control Samples?
- c. Blanks?

19.2 Are the VOA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (quant. reports) included in the sample package for each of the following:

- a. Samples and/or fractions as appropriate?
- b. Laboratory Control Samples
- c. Blanks?

**ACTION:** If any data are missing, take action specified in 1.1 above.

19.3 Is chromatographic performance acceptable with respect to:

- a. Baseline stability?
- b. Resolution?
- c. Peak shape?
- d. Full-scale graph (attenuation)?
- e. Other: \_\_\_\_\_?

**ACTION:** Use professional judgement to determine the acceptability of the data.

19.4 Are the lab-generated standard mass spectra of the identified compounds present for each sample?

**ACTION:** If any mass spectra are missing, take action as specified in 1.1 above. If the lab does not generate its own standard spectra, document in the Contract Problems/Non-compliance section of the Data Assessment.

19.5 Is the RRT of each reported compound within 0.06

YES NO N/A

RRT units of the standard RRT in the continuing calibration?

19.6 Are all ions present in the reference standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum?

19.7 Do sample and reference standard relative ion intensities agree within ±20%?

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected "R", flagged "N" (presumptive evidence of the presence of the compound) or changed to not detected "U" at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in 19.5, 19.6, and 19.7

20.0 Field Duplicates

20.1 Were any field duplicates submitted for VOA analysis?

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Note the RPD value in the data assessment.

This Data Assessment is based on USEPA Region II SOP HW- : Volatile Organics Analysis of Ambient Air in Canisters by Method TO-15, May 2004.

Case No. \_\_\_\_\_ SDG No. \_\_\_\_\_ LABORATORY: \_\_\_\_\_

SITE : \_\_\_\_\_

All data are valid and acceptable except those analytes which have been qualified with a "J" (estimated), "U"(non-detects), "R" (unusable), or "N" (presumptive). All action is detailed on the following sheets.

The following facts should be noted by all data users. First, the "R" flag means that the associated value is unusable. In other words, due to s Significant QC problems, the analysis is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on data tables because they cannot be relied upon, even as a last resort. The second fact to keep in mind is that no compound concentration, even if it has passed all QC tests, is guaranteed to be accurate. Strict QC serves o to increase confidence in data but any value potentially contains error. In addition the "N" flag shows that the analysis indicates the presence of an analyte for which there is presumption evidence to make a "tentative identification."

All actions are detailed below and on the attached sheets:

Overall Assessment:

Contract Non-Compliance:

YES NO N/A

Reviewer's  
Signature: \_\_\_\_\_ Date: \_\_\_\_/\_\_\_\_/20\_\_

Verified By: \_\_\_\_\_ Date: \_\_\_\_/\_\_\_\_/20\_\_