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Review of the methods of the US Environmental Protection Agency for bromate determination and validation of Method 317.0 for disinfection by-product anions and low-level bromate

Daniel P. Hautman[a,*], David J. Munch[a], Christopher Frebis[a], Herbert P. Wagner[b], Barry V. Pepich[b]

[a]*US Environmental Protection Agency, Office of Ground Water and Drinking Water, 26 W. Martin Luther King Dr., Cincinnati, OH 45268, USA*

[b]*IT Corporation, Technology Applications Group, 26 W. Martin Luther King Dr., Cincinnati, OH 45219, USA*

Abstract

In recent years several methods have been published by the United States Environmental Protection Agency (EPA) which specify bromate as a target analyte. The first of these was EPA Method 300.0. As technological improvements in ion chromatographic hardware have evolved and new detection techniques have been designed, method detection limits for bromate have been reduced and additional procedures have been written, including EPA Method 300.1, 321.8 and, most recently, EPA Method 317.0. An overview of the evolution of these bromate methods since 1989 is presented. The focus is specific to each of these respective procedures, highlighting method strengths, weaknesses, and addressing how these methods fit into EPA's regulatory agenda. In addition, performance data are presented detailing the joint EPA/American Society for Testing and Materials multilaboratory validation of EPA Method 317.0 for disinfection by-product anions and low-level bromate. © 2001 Published by Elsevier Science B.V.

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1. Introduction

Bromate has been identified as an inorganic disinfection by-product (DBP) in public water supplies (PWS) following ozonation [1,2]. Bromate has also been identified as an animal carcinogen [3] and has been classified as a group 2B, probable human carcinogen by the International Agency for Research on Cancer [4]. Health effects research indicates it to be a suspected human carcinogen which exhibits a potential 10^{-4} risk of cancer after a lifetime exposure in drinking water at 5.0 μg/l and a potential 10^{-5} risk at 0.5 μg/l [5].

In August 1993, EPA published Method 300.0, revision 2.1 [6]. Method 300.0 was the first EPA ion chromatographic (IC) analytical method and remains widely accepted as the standard EPA IC method for common anions. In September 1997, EPA published Method 300.1 [7]. Method 300.1 applied the basic IC principles outlined in Method 300.0 but redefined the operating conditions to enable the quantitation of significantly lower concentrations of bromate and specified quality control (QC) criteria which went beyond those included in Method 300.0.

Further analytical method development work was completed on bromate within the Office of Research and Development (ORD), National Exposure Research Laboratory (NERL) which published EPA

*Corresponding author.

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Method 321.8 in December 1997 [8]. This method involves the ion chromatographic separation of bromate followed by detection using inductively coupled argon plasma (ICAP) mass spectrometry (MS) and has been reported as both a very selective and sensitive procedure [9,10].

In December 1998, EPA promulgated a maximum contaminant level (MCL) for bromate in drinking water under Stage 1 of the Disinfectants/Disinfection By-products (D/DBP) Rule [11]. The current bromate MCL is 10 μg/l [11]. At the same time, the maximum contaminant level goal (MCLG) for bromate was set at zero under Stage 1 of the D/DBP Rule [11]. The limitation of available compliance monitoring methods for trace bromate was one of the factors in establishing the Stage 1 D/DBP bromate MCL at 10 μg/l.

Between September 1998 and the spring of 2000, EPA presented and published method development work designed to improve trace bromate measurement using a postcolumn reagent, *o*-dianisidine dihydrochloride (ODA). The postcolumn absorbance detection system is connected in series, directly to the instrument configuration found in EPA Method 300.1 [12–15]. This development work has been subjected to an interlaboratory validation study, a complete peer review, and has been published as EPA Method 317.0 [16].

This article elucidates some of the similarities and differences between EPA Methods 300.0, 300.1, 321.8 and 317.0, all of which specify bromate in their target analyte list. The capabilities and performance of these methods are examined and their application to fulfilling a regulatory role in compliance monitoring for bromate is also addressed. In addition, a summary of the statistical data collected during the interlaboratory validation of Method 317.0 is presented.

2. Experimental

2.1. EPA Method 300.0

Method 300.0 was originally developed in 1989 but the most recent publication of the method was revision 2.1, August 1993. The method is divided into Part A for common anions and Part B for the oxyhalides. The specific conditions prescribed by this method are presented in Table 1. Basically, this method specifies a carbonate-based eluent, requires unique analytical columns for Part A (common anions) and Part B (oxyhalides), and uses suppressed conductivity detection.

2.2. EPA Method 300.1

The development of Method 300.1 was largely driven by the proposed Stage 1 D/DBP Rule [5] regulatory requirement to quantitate bromate at 10 μg/l in drinking water. The bromate minimum detection limit (MDL) [17], as defined in Method 300.0, was too high to support a bromate MCL at 10 μg/l. Therefore, Method 300.1 was developed as a more sensitive method by identifying specific parameters (column, eluent and injection volume) which could be utilized to provide quantitation of lower concentrations of bromate in drinking water, even in the presence of up to 50 mg/l chloride. This method incorporated an IC column with higher capacity, capable of more efficiently resolving trace bromate from the common anions in field samples, particu-

Table 1
EPA Method 300.0 analytical conditions

Method parameter	Method specification
Analytical column	Part A: Dionex AG4A and AS4A, 4 mm (or equivalent) Part B: Dionex AG9 and AS9, 4 mm (or equivalent)
Eluent	1.7 m*M* sodium hydrogencarbonate–1.8 m*M* sodium carbonate
Eluent flow-rate	2.0 ml/min
Detection	Suppressed conductivity
Sample injection volume	Part A and Part B: 50 μl

larly when injecting relatively large volumes (200 μl) of complex sample matrices. The analytical conditions specific to Method 300.1 are presented in Table 2.

While these modifications were performed primarily to reduce the quantitation level for bromate, it was decided that it would be more complete to include all the Method 300.0 analytes in Method 300.1. Additionally, the hardware requirements specified in Method 300.0 were simplified in Method 300.1 by specifying a single analytical column for both Part A and Part B.

By closely examining Section 2.4 of Method 300.0 regarding method modifications, and Sections 6.2.2.1 and 6.2.2.2 regarding optional analytical columns, it becomes clear that Method 300.1 is completely embraced by the guidelines in Method 300.0. A laboratory can adopt the parameters, columns and specifications identified in Method 300.1, fulfill all the required QC criteria shown in Section 9 of Method 300.0, and legitimately claim that they are using Method 300.0 (modified as permitted in the above referenced sections) when applying for state certification.

Shortly after the development of EPA Method 300.1, the American Society of Testing and Materials (ASTM), D19.05 Task Group began to consider adopting many of the conditions identified in EPA Method 300.1 as an ASTM standard method. In July 2000, ASTM D6581-00 was approved and is expected to be published in September 2000 [18]. One significant contribution in this ASTM D6581-00 standard is the interlaboratory precision and bias data that were generated for this method. These data were comparable to those data EPA collected for the interlaboratory validation of Method 317.0 on the suppressed conductivity detector.

Table 2
EPA Method 300.1 analytical conditions

Method parameter	Method specification
Analytical column	Part A and Part B: Dionex AG9-HC and AS9-HC, 2 mm OR 4 mm (or equivalents)
Eluent	9.0 m*M* sodium carbonate
Eluent flow-rate	0.40 ml/min (2 mm column) OR 1.25 ml/min (4 mm column)
Detection	Suppressed conductivity
Sample injection volume	Part A: 10 μl (2 mm column) OR 40 μl (4 mm column) Part B: 50 μl (2 mm column) OR 200 μl (4 mm column)

2.3. EPA Method 321.8

The specific analytical conditions for EPA Method 321.8 are presented in Table 3. This method was developed at nearly the same time as Method 300.1 and represents a procedure that can provide a very selective and sensitive response for low-level bromate. This IC–ICAP-MS method is unique and is not closely related to any of the other three methods (300.0, 300.1 and 317.0) discussed in this article. The ICAP-MS system operates by ionizing all bromine-containing species in the ICP argon plasma, as they elute from the analytical column. These ions are then evaluated at 79 and 81 atomic mass units (u) by an interfaced mass spectrometer. Since the eluting ions are monitored at both 79 u and the bromine isotope fraction at 81 u, the analyst can also monitor for the later eluting bromide anion (Br^-) in the same analysis, although the bromide anion is not listed in the method's target analyte list.

2.4. EPA Method 317.0

With the original publication of EPA Method 300.0 and subsequent release of EPA Method 300.1, the continued interest in improving method sensitivity for bromate led to the development of EPA Method 317.0, which is directly related to EPA Method 300.1. The specific analytical conditions for EPA Method 317.0 are presented in Table 4. EPA Method 317.0 was built directly onto the existing Method 300.1 instrument configuration to further increase the sensitivity for trace bromate analysis and simultaneously maintain all the capabilities of Method 300.1 for conductivity detection. By taking the effluent stream exiting the conductivity cell, removing the backpressure coil(s) (which are specified when using a Dionex electrolytic suppressed conductivity system) and redirecting this suppressed eluent to a postcolumn reagent (PCR) mixing and detection system, a very selective and sensitive method of detecting trace bromate was realized. This

Table 3
EPA Method 321.8 analytical conditions

Method parameter	Method specification
Analytical column	Dionex PA-100 (guard and analytical), 4 mm (or equivalent)
Eluent	5.0 m*M* nitric acid–25 m*M* ammonium nitrate
Eluent flow-rate	1 ml/min
Detection	Suppressed eluent, nebulized to the inductively coupled plasma (ICP) MS
Sample injection volume	580 μl
Additional method defined chromatographic parameters specific to EPA 321.8	
Pretreatment cartridge	Dionex On-Guard RP (or equivalent)
Drift standard loop	170 μl
Additional method defined ICP-MS parameters specific to EPA Method 321.8	
Power	1.4 kW
Cool gas	12.0 l/min
Aux gas	1.2 l/min
Nebulizer gas	0.957 l/min (concentric)
m/*z* monitored	79 and 81
Analysis mode	Time resolved or chromatographic
Time slice	0.40 s
Spray chamber	5°C
Sensitivity (100 μg/l bromate)	35 000 cps *m*/*z* 79
Background (of eluent)	100 cps (*m*/*z* 79; 2500 cps *m*/*z* 81)

Table 4
EPA Method 317.0 analytical conditions

Method parameter	Method specification
Analytical column	Dionex AG9-HC and AS9-HC, 4 mm only (or equivalents)
Eluent	9.0 m*M* sodium carbonate
Eluent flow-rate	1.3 ml/min (since ONLY the 4 mm column can be used)
Detection	Suppressed conductivity followed in series with PCR absorbance detector
Sample injection volume	225 μl
Additional method defined parameters specific to EPA Method 317.0	
PCR reagent flow	0.70 ml/min
PCR reagent preparation (careful attention must be paid to the required purity of these reagents, as defined in the method)	Step 1: to 300 ml reagent water add: 40 ml 70% nitric acid and 2.5 g KBr Step 2: dissolve 250 mg *o*-dianisidine dihydrochloride (purified) in 100 ml methanol, with stirring Step 3: mix both solutions and dilute to final volume of 500 ml Step 4: allow time for PCR mix to clarify
Postcolumn reactor coil	Potted, knitted, 500 μl internal volume, securely held in heater
Postcolumn heater temperature	60°C
Absorbance detector flow cell	10 mm path length
Absorbance lamp	Tungsten
Wavelength setting	450 nm

PCR system was designed for simplicity and includes a single PCR solution reservoir, mixing tee, heated reaction coil and an absorbance detector set to 450 nm.

2.4.1. Joint EPA/ASTM interlaboratory validation of EPA Method 317.0

An interlaboratory validation study was conducted as a means to both investigate the adaptability of the PCR method to laboratories with comparable instrumentation and to assess interlaboratory precision and bias [19]. Twelve laboratories were solicited to participate in the validation study. Seven laboratories responded favorably with an interest in participating and data were provided by five of these laboratories. Prior to statistical evaluation, the data from the five laboratories were subjected to Dixon's test for outlying observations [20].

In order to eliminate the potential for bias introduced as a result of fortification errors in the various participating laboratories, all samples were prepared, packaged and sent to the participants ready for direct analysis. The analysis array for this validation study required laboratories to complete a determination of MDLs for both suppressed conductivity and PCR absorbance detection, as well as a series of several fortified study matrices, which had been prepared as Youden pairs [21]. The Youden ''paired sample'' approach to collaborative testing and data analysis [21] was incorporated in this study to evaluate interlaboratory precision and bias for Method 317.0. This approach uses ''pairs'' of fortified samples at slightly different concentrations, rather than replicate levels, to reduce the risk of analyst interpretive bias. Calibration curves were established for the anions chlorite, bromide, chlorate and bromate (above 10 μg/l) measured by suppressed conductivity detection and trace bromate (between 0.50 and 15 μg/l) by PCR absorbance detection at 450 nm. These anions were studied in three sample matrices, preserved as defined in the method; reagent water, bottled (ozonated) water and chlorinated tap water. The actual concentration range of the anions evaluated on the conductivity detector in each matrix were chlorite at 108–357 μg/l, bromate at 11–31 μg/l, bromide at 36–187 μg/l and chlorate at 72–549 μg/l. On the PCR absorbance detector, trace bromate was exclusively monitored in the test matrices at concentrations between 1.5 and 5.1 μg/l.

3. Results and discussion

3.1. Reagent water method detection limits

The MDLs [20] for bromate are shown in Table 5 for Methods 300.0, 300.1, 321.8 and 317.0 (both by suppressed conductivity and by PCR absorbance). From this table, it is apparent that Method 321.8 and the PCR absorbance detection of bromate in Method

Table 5
Reagent water bromate detection limits

Method	Detector type	Fort. conc. (μg/l)	*n*	MDL[a] (μg/l)	Expected MRL (μg/l)
300.0	Suppressed cond.	NDR[b]	7	20	60[c]
300.1	Suppressed cond.	2.0	7	1.4	5.0[d]
321.8	ICP-MS	NDR[b]	7	0.30	0.90[c]
317.0	Suppressed cond.	2.0	8	0.71	5.0[d]
	PCR absorbance	0.50	7	0.12	0.50[d]

[a] MDL, minimum detection limit is the minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero [20]. This is NOT the minimum reporting level (MRL) which is the minimum concentration that can be reported as a quantitated value for a target analyte in a sample following analysis. The MRL for a given laboratory, as defined in Methods 300.1 and 317.0, can be no lower than the concentration of the lowest calibration standard and can only be used if acceptable quality control criteria for this lowest standard, as the initial calibration check standard, are met.

[b] NDR, no data reported for fortified concentration in the method.

[c] Estimated MRL using a multiplier of three times the published MDL concentration.

[d] MRL routinely used at the Office of Ground Water and Drinking Water, Technical Support Center laboratory.

317.0 have the lowest MDLs. It should be noted, however, that the calculated MDL should primarily be used as a gauge to assist in approximating the laboratory's minimum reporting level (MRL). The MRL is the minimum concentration that can be reported as a quantitated value for a target analyte in a sample following analysis [7,16]. The MRL for a given laboratory, as defined in Methods 300.1 and 317.0, can be no lower than the concentration of the lowest calibration standard and can only be used if acceptable quality control criteria for this lowest standard, used as the initial calibration check standard (ICCS), are met.

3.2. Precision and recovery assessment

Each EPA bromate method contains precision [defined as the relative standard deviation (RSD), expressed as a percent] and recovery data from replicate analyses of fortified samples prepared in various matrices. These single laboratory precision and recovery data, exclusively for bromate and as published in each respective method, are presented in Table 6.

Methods 300.0, 300.1 and 317.0 contain data for fortified 18 MΩ reagent water (RW) and fortified tap water (TW) at various prepared concentrations. The

Table 6
Single laboratory precision and recovery for bromate as published in the respective method

Method	Detector type	Matrix type	Fort. conc. (μg/l)	*n*	Mean recovery (%)	RSD of mean recovery (%)
300.0	Suppressed cond.	RW[a]	50	7	122	20
		TW[b]	50	7	98.0	10
		O_3W[c]	NDR[d]	–	–	–
300.1	Suppressed cond.	RW	5.0	9	101	8.9
			25	9	106	6.5
		TW	5.0	9	93	17
			25	9	100	6.6
		O_3W	5.0	9	81	11
			25	9	91	4.7
321.8	ICAP-MS	RW	NDR	–	–	–
		TW	NDR	–	–	–
		O_3W	MC[e] 0.80 μg/l	5	–	16[f]
			25	5	102	2.4
		O_3W	MC[e] 3.0 μg/l	5	–	6.4[f]
			25	5	98	1.4
		O_3W	MC[e] 10 μg/l	5	–	3.6[f]
			25	5	98	3.4
317.0	Suppressed cond.	RW	5.0	8	96	8.8
		TW	5.0	8	82	16
		O_3W	NDR	–	–	–
	PCR absorbance	RW	0.50	8	100	10.8
			5.0	8	108	2.1
		TW	0.50	8	106	2.8
			5.0	8	104	1.9
		O_3W	NDR	–	–	–

[a] RW, reagent water.
[b] TW, tap water, either chlorinated or defined as simply ''drinking water'' in method.
[c] O_3W, ozonated tap water.
[d] NDR, no data reported for this type of matrix in the method.
[e] MC, matrix concentration, as the mean concentration measured in the unfortified sample.
[f] The values represent the % RSD of the mean concentration measured in the unfortified sample.

fortified precision and recovery data published in Method 300.0 were determined at a fortification level of 50 μg/l and were generally low, reflecting 98 to 122% recovery and a precision of 10 to 20% (expressed as the RSD in the mean recovery). However, these results are expected since the published MDL for bromate is 20 μg/l. For Method 300.1, at a fortified concentration of 25 μg/l, recoveries are very good and fall in a range of 91 to 106% with precision between 4.4 and 6.6% RSD. When the fortified concentration was reduced to 5.0 μg/l, the recoveries begin to deteriorate and extend from 81 to 101% with corresponding precision falling from between 8.9 and 17% RSD. For Method 317.0, RW and TW fortified samples were prepared at 5.0 and 0.50 μg/l. The recoveries for these samples were excellent, ranging from 100 to 108% with a corresponding precision from 1.9 to 10.8% RSD.

Method 321.8 has data exclusively for an ozonated water (O_3W) matrix with replicate data for both the unfortified samples and the same sample matrices fortified at 25 μg/l. These Method 321.8 data reflect a high degree of precision, even down to measured native bromate concentrations of 0.80 μg/l.

The bromate fortification levels for methods 300.0, 300.1 (at 5.0 μg/l) and 317.0 were all within a factor of 4 of their respective published MDLs and were, in most cases, at or near the expected MRLs shown in Table 5. This low-level fortification data is useful to estimate precision near the laboratory MRL. Method 321.8 used replicate analyses of various ozonated sample matrices which had a native concentration for bromate observed near what would likely approach the laboratory's expected MRL.

3.3. EPA Method 317.0 validation study

3.3.1. Interlaboratory determined method detection limits

At each of the five laboratories, the conductivity MDLs were determined using seven replicate analyses containing 5.0 μg/l chlorite, bromate, bromide and chlorate in reagent water and the bromate PCR absorbance detector MDL was determined by analyzing seven replicates of a 1.0 μg/l bromate addition in reagent water. One laboratory's MDL data, for the bromate measurement by PCR absorbance, was classified as an outlier and the data were rejected prior to statistical evaluation. These results are presented in Table 7.

3.3.2. Interlaboratory average precision and bias

Table 8 contains precision and bias summary data following the Method 317.0 interlaboratory validation study. For chlorite (108–357 μg/l), bromate (11–31 μg/l), bromide (36–187 μg/l) and chlorate (72–549 μg/l) by conductivity detection, the data reflected an average single analyst precision of 10% RSD (0.6 to 17%), an average interlaboratory precision of 12% RSD (2.5 to 25%) and an average bias of 1.6% (−9.0 to 16%). For the trace level analysis

Table 7
Method 317.0 interlaboratory validation study[a] MDLs (μg/l)

Laboratory	Chlorite MDL (cond[b])	Bromide MDL (cond)	Bromate MDL (cond)	Chlorate MDL (cond)	Bromate MDL (PCR)
1	1.05	1.51	1.48	1.04	0.16
2	2.47	2.92	1.22	3.95	0.19
3	1.27	2.24	1.93	2.07	0.41
4	1.20	1.45	4.07	3.14	0.18
5	2.13	5.77	2.15	4.90	0.98[c]
Average	1.62	2.78	2.17	3.02	0.24
SD	0.63	1.78	1.12	1.52	0.12

[a] Complete details of the validation study report presented in Ref. [18].
[b] ''cond'' indicates suppressed conductivity.
[c] Rejected using Dixon's outlier test [18].

Table 8
Method 317.0 validation study[a] summary of results, average precision and bias

Analyte	Bias (%) (range)	Single analyst precision (RSD, %) (range)	Interlaboratory precision (RSD, %) (range)
Bromate (cond[b])	0.35 (−7.8 to 5.1)	10 (3.7 to 17)	12 (4.6 to 25)
Chlorite	−0.98 (−9.0 to 2.6)	1.9 (0.6 to 4.1)	4.2 (2.5 to 7.8)
Bromide	−0.87 (−6.0 to 3.7)	4.2 (0.6 to 9.6)	6.9 (4.7 to 14)
Chlorate	1.6 (0.30 to 6.0)	2.4 (0.7 to 6.1)	4.5 (2.8 to 6.0)
Bromate (PCR)	4.8 (−0.20 to 16)	7.3 (1.2 to 10)	9.6 (3.2 to 16)

[a] Complete details of the validation study report presented in Ref. [18].
[b] ''cond'' indicates suppressed conductivity.

of bromate (1.5–5.1 μg/l) by the PCR absorbance detector, the data reflected an average single analyst precision of 8.0% RSD (1.2 to 10%), an average interlaboratory precision of 10% RSD (3.2 to 16%) and an average bias of 5.0% (−0.20 to 16%).

4. Conclusions

EPA Method 300.0 is regarded as the standard method for common anion analysis, but due to limitations with regard to sensitivity, the method has limited utility for low-level bromate analysis and consequently, for this oxyhalide anion, cannot be used for regulatory support. EPA Method 300.1 specifies the use of a single, high capacity analytical column that is capable of monitoring bromate, as well as the other anions listed in the method. The Method 300.1 prescribed column has a higher ion-exchange capacity which improves chromatographic resolution and minimizes the potential for chromatographic interferences from the common anions at concentrations up to 10 000 times greater than the bromate anion. Minimizing the interferences allows the injection of a larger sample volume, which can yield MDLs in the range of 1–2 μg/l. While these detection limits were certainly better than those specified in Method 300.0, the lowest practical MRL a laboratory could likely achieve, without significant further technological improvements to the method, would be approximately 5 μg/l. For drinking water systems interested in carefully evaluating treatment processes and precisely monitoring bromate concentrations below 10 μg/l, accurate quantitation will be difficult and can reflect poor precision.

During the mid-1990s, the EPA Office of Research and Development (ORD), National Exposure Research Laboratory (NERL) also began to investigate alternate methods for detecting low levels of bromate. The IC–ICAP-MS procedure utilizes IC separation of the bromate fraction of a sample matrix, and then ionizes this eluting bromate in the argon plasma. The resulting ions are then transferred to a mass spectrometer where amu 79 and 81 are monitored. This means of detection is both sensitive and selective for any bromine-containing species that will chromatographically separate on the IC system. Method 321.8 is currently being considered for bromate monitoring in the proposal for the Stage 2 D/DBP rule. Although the method is capable of selectively detecting low levels of bromate, the capital investment for equipment and high level of skill required to perform the analysis may result in limited application of this procedure.

As a way to utilize existing IC instrumentation and simplify the analyses, EPA recently adapted the existing hardware configuration specified in Method 300.1, and directly interfaced an additional detection system, in series, immediately following the suppressed conductivity detection. Published as EPA Method 317.0, this method uses a postcolumn reagent that reacts with the eluting bromate to form a chromophore which is then measured using an absorbance detector. Acceptable precision and recovery of bromate at both 5.0 μg/l and the trace level of 0.5 μg/l have been demonstrated. This method was further evaluated in an interlaboratory validation study and these results indicated that the method has widespread applicability. Method 317.0 provides excellent bromate sensitivity and offers a

practicable method for future compliance monitoring for all of the inorganic oxyhalide DBPs as well as trace levels of bromate. Method 317.0 will allow detection of bromate below the MCL of 10 μg/l and is currently being considered for bromate monitoring in the proposal for the Stage 2 D/DBP rule.

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