



Designation: D8027 – 17

Standard Practice for Concentration of Select Radionuclides Using MnO₂ for Measurement Purposes¹

This standard is issued under the fixed designation D8027; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ε) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This practice is intended to provide a variety of approaches in which manganese oxide (MnO₂) can be used to concentrate radionuclides of interest into a smaller volume counting geometry or exclude other species that would otherwise impede subsequent chemical separation steps in an overall radiochemical method, or both.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

[D1129 Terminology Relating to Water](#)

[D7902 Terminology for Radiochemical Analyses](#)

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this standard, refer to Terminologies [D1129](#) and [D7902](#).

¹ This practice is under the jurisdiction of ASTM Committee [D19](#) on Water and is the direct responsibility of Subcommittee [D19.04](#) on Methods of Radiochemical Analysis.

Current edition approved June 1, 2017. Published June 2017. Originally approved in 2016. Last previous edition approved in 2016 as D8027 – 16. DOI: 10.1520/D8027-17.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

4. Summary of Practice

4.1 These practices describe different processes through which MnO₂ can be used to concentrate specific radionuclides of interest into a smaller volume counting geometry or exclude other species that would otherwise impede subsequent chemical separation steps in an overall radiochemical method, or both.

4.2 Published studies **(1-5)**³ have addressed in detail the various manners in which hydrous manganese dioxides can be synthesized and the variety of crystal forms of hydrous manganese dioxide that can result. The literature describes the following general categories in which hydrous manganese dioxide can be prepared.

4.2.1 *Guyard Reaction:*



4.2.2 By the reduction of permanganate with reducing reagents such as hydrogen peroxide (H₂O₂) or hydrogen chloride (HCl).

4.2.3 By the oxidation of Mn(II) salt under alkaline conditions with oxidizing reagents such as potassium chlorate (KClO₃), H₂O₂, ozone (O₃), or ammonium persulfate ((NH₄)₂S₂O₈).

4.3 The presented practices are not meant to address every possible approach to the generation and use of MnO₂ but are meant to present some more typical practices that may be generally useful.

5. Significance and Use

5.1 This practice is applicable to the separation of specific radionuclides of interest as part of overall radiochemical analytical methods. Radionuclides of interest may need to be quantified at activity levels of less than 1 Bq. This may require measurement of less than 1 fg of analyte in a sample which has a mass of a gram to more than several kilograms. This requires concentration of radionuclides into a smaller volume counting geometry or exclusion of species which would impede subsequent chemical separations, or both. MnO₂ has shown good

³ The boldface numbers in parentheses refer to a list of references at the end of this standard.

selectivity in being able to concentrate the following elements: actinium (Ac), bismuth (Bi), lead (Pb), polonium (Po), plutonium (Pu), radium (Ra), thorium (Th), and uranium (U) as noted in the referenced literature (see Sections 4 and 8). The MnO_2 can be loaded onto a variety of substrates in preparation for use or generated in-situ in an aqueous solution. The presented processes are not meant to be all encompassing of what is possible or meant to address all limitations of using MnO_2 . Some limitations are noted in Section 6, Interferences.

6. Interferences

6.1 MnO_2 is able to achieve a very good decontamination factor from monovalent cations in solution as evidenced by 8.5 below in which it is used in seawater. However, in the case of elevated concentrations of divalent cations, for example barium, the recovery of analytes of interest can be significantly reduced (6). Additionally in the case of seawater, the recovery of analytes such as uranium may also be substantially reduced. In such cases the use of an isotopic tracer can be very important to correct for such reduced recovery. The MnO_2 separation is also very conducive to being easily repeated to achieve a second stage of separation from potentially interfering species.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ Other grades may be used, provided that the reagent is of sufficiently high purity to permit its use without increasing the background of the measurement.

7.1.1 Some reagents, even those of high purity, may contain naturally occurring radioactivity, such as isotopes of uranium, radium, actinium, thorium, rare earths and potassium compounds or artificially produced radionuclides, or both. Consequently, when such reagents are used in the analysis of low-radioactivity samples, the activity of the reagents shall be determined under analytical conditions that are identical to those used for the sample. The activity contributed by the reagents may be considered to be a component of background and applied as a correction when calculating the test sample result. This increased background reduces the sensitivity of the measurement.

7.2 *Ammonium hydroxide, 15 M NH_4OH* , (concentrated reagent).

7.3 *Ammonium hydroxide, 6 M NH_4OH* —Add 400 mL of concentrated ammonium hydroxide to 400 mL water. Dilute to 1 L with water and mix well.

7.4 *Bromocresol purple pH indicator*—Add 0.1 g bromocresol purple in 18.5 mL of 0.01 M sodium hydroxide (NaOH) solution. Dilute to 250 mL with water and mix well.

⁴ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the United States *Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

7.5 *Hydrogen peroxide, 30 % H_2O_2* —The ACS specification allows for a concentration range of 29 to 32 %.

7.6 *Iron chloride, FeCl_3* .

7.7 *Potassium permanganate, KMnO_4* .

7.8 *Potassium permanganate, 0.5 M KMnO_4* —Add 79 g of KMnO_4 to 750 mL water. Dilute to 1 L with water and mix well.

7.9 *Sodium hydroxide, 0.01 M NaOH*—Add 0.1 g NaOH to 250 mL water.

7.10 *Phenolphthalein pH indicator solution*—Commercially available as 1 % solution in ethanol.

7.11 *Sodium hydroxide, 10 M*—Carefully dissolve 400 g of NaOH in about 900 mL of water and dilute to 1 L.

7.12 *Manganese (II) chloride, 0.5 M*—Dissolve 11.7 g of $\text{MnCl}_2 \cdot 6\text{H}_2\text{O}$ in about 80 mL of water and dilute to 100 mL.

8. Procedure

8.1 *Use of MnO_2 Generated in-situ to Pre-Concentrate Sample Analytes:*

8.1.1 The precipitation of MnO_2 from a water sample may be most conveniently performed on a volume of 0.1 to 2 L but larger volumes are possible (7 and 8). Any isotopic tracers should be added and the valence states of the tracers and analyte species allowed to equilibrate before proceeding.

8.1.2 Add to the water sample approximately 10 mg of KMnO_4 and allow to dissolve. Optionally, approximately 2 mg of FeCl_3 may also be added to improve the obtainable separation factor.

8.1.3 The pH indicator bromocresol purple may be added to the water sample to provide a color indicator in the following step of raising the pH.

8.1.4 Add to the water sample approximately 1 mL of about 30 % H_2O_2 .

8.1.5 The precipitation step to follow is best performed at ambient temperature if a period of one or more days is available to allow for complete settling and development of the precipitate. Alternatively the promptness of the precipitation can be assisted by gently heating the water sample, for example to approximately 70–80°C.

8.1.6 Add sufficient 6 M NH_4OH to the water sample to raise the pH to about 8. If bromocresol purple was added in the prior step this pH would be indicated by a color change to purple. The needed pH change could also be measured through use of pH test strip or pH meter.

8.1.7 Following complete development and settling of the precipitate the supernate may be removed by aspiration or careful decantation. The small amount of MnO_2 precipitate may be optimally isolated into a small pellet by transferring the bottom layer containing the precipitate (about 50 mL) to a centrifuge tube and centrifuging. The precipitate may be further washed up to two times with water and centrifugation repeated.

8.1.8 An alternative to centrifugation is filtration through a 0.45 μm filter but may require more time to accomplish the filtration in a careful manner.

NOTE 1—A similar procedure can be used on acid leachates of sediment

samples but care should be taken when adding 6 M NH_4OH to strong acid solutions.

8.1.9 The washed MnO_2 precipitate and the incorporated analytes of interest and associated isotopic tracers may be taken through further separative steps, for example extraction chromatography, or may be transferred to a suitable counting geometry.

8.2 *In-situ MnO_2 Precipitation by Oxidation of Mn(II) using H_2O_2 :*

8.2.1 An example of forming an in-situ MnO_2 precipitate by oxidation of Mn(II) salt using H_2O_2 is given in this section. This example has been used to pre-concentrate radium from 0.5 L aqueous samples for the determination of ^{224}Ra and ^{226}Ra by alpha spectrometry.

8.2.2 Add an appropriate amount of ^{225}Ra tracer to the sample aliquant.

8.2.3 Acidify the sample using nitric or hydrochloric acid. An approximate acid concentration of 0.1 to 0.2 M is suggested.

8.2.4 Adjust the pH of the sample using phenolphthalein pH indicator and 10 M NaOH to produce the first sustained pink color and then add a few additional drops of 10 M NaOH to provide a basic solution with a pH between 10 and 12. Perform the pH adjustment and the following steps while continuously stirring on a stir plate with a magnetic stir bar.

8.2.5 Add 1 mL of 0.5 M Manganese (II) chloride and mix well.

8.2.6 Add approximately 2 mL of 30 % H_2O_2 using a plastic transfer pipette.

8.2.7 MnO_2 precipitate (blackish color) will rapidly form and effervescence occurs.

8.2.8 Continue stirring until effervescence subsides and then allow the precipitate to settle (remove the magnetic stir bar).

8.2.9 Most of the supernatant is removed by aspiration or decantation after the precipitate has settled. Transfer the remaining suspension to a centrifuge tube and isolate the MnO_2 precipitate by centrifuging and decanting the supernatant. Alternatively if a more rapid analysis is required the MnO_2 may be isolated immediately after precipitation is complete by centrifuging in a large volume centrifuge bottle (this eliminates the time required for settling).

8.2.10 It may be advantageous to reduce the amount of calcium retained by the MnO_2 precipitate. For example the determination of ^{224}Ra and ^{226}Ra by alpha spectrometry can be adversely affected by excessive calcium. If calcium levels are too high CaSO_4 will precipitate during the Ba/RaSO_4 micro-precipitation step and spectral quality will be degraded. The following steps describe an approach for reducing calcium retained on the MnO_2 precipitate.

8.2.10.1 Dissolve MnO_2 precipitate which has been collected in a centrifuge tube with 5 mL of 2 M HCl and about 0.1 mL of 30 % H_2O_2 . Cap the tube and shake vigorously to facilitate dissolution.

8.2.10.2 Dilute to about 40 mL and add about 0.1 mL of 30 % H_2O_2 .

8.2.10.3 Re-precipitate MnO_2 by adding approximately 2 mL of concentrated NH_4OH with a plastic transfer pipette. Cap tube and mix gently. MnO_2 precipitate will form and efferves-

cence will occur. Loosen cap to vent gas. Tighten cap and gently mix several times over a period of several minutes until the precipitate settles and a clear supernatant appears.

8.2.10.4 Centrifuge the suspension and decant the supernatant. The supernatant will contain a significant fraction of the calcium which was retained on the original MnO_2 precipitate (thus reducing the amount of calcium retained by the MnO_2).

8.2.10.5 The MnO_2 re-precipitation described above can be repeated if necessary to further reduce calcium retained on the MnO_2 precipitate.

8.3 *Screening for Naturally Occurring Radionuclides using MnO_2 :*

8.3.1 MnO_2 freshly precipitated from aqueous samples effectively retains a variety of naturally occurring radionuclides such as radium, thorium, uranium, polonium, actinium, and lead. Sodium and calcium are much more weakly retained. Freshly precipitated MnO_2 could provide a useful approach for screening for naturally occurring alpha and beta emitters in samples with high levels of sodium and calcium. Larger volumes could be analyzed since most of the sodium and calcium are not retained and the mass of the MnO_2 precipitate is much less than the mass of salts obtained by evaporation. Useful techniques for preparing a counting source from a MnO_2 precipitate could include:

8.3.1.1 Direct collection by filtration.

8.3.1.2 Dissolution with nitric acid and H_2O_2 followed by evaporation on a stainless steel planchet.

8.3.1.3 Dissolution with dilute acid and H_2O_2 and then mixing with liquid scintillation cocktail.

8.4 *Use of MnO_2 Impregnated Resin Beads:*

8.4.1 A typical radiochemical separation geometry is the use of a column into which an aqueous sample can be poured and excellent contact made with a supported extraction media. MnO_2 has also been found to be quite amenable to this approach (9-13). MnO_2 loaded resin can be made or purchased commercially for use in such column geometry. This approach allows the amount of MnO_2 loaded resin to be readily adjusted to the sample aqueous volume and expected analyte concentration. As well the retained analytes can also be readily eluted from the supported media using the same column geometry.

8.5 *Use of MnO_2 Impregnated Polymer Fibers/Cartridges:*

8.5.1 The naturally occurring alpha-emitting radium isotopes ^{223}Ra , ^{224}Ra , and ^{226}Ra as well as beta-emitting ^{228}Ra have been extensively used as geochemical tracers in studies of large water bodies – primarily seawater but fresh water as well. Such studies rely on the large variation in half-lives of those radium isotopes – from a few days to more than a thousand years to characterize the sources of those radium isotopes and their mixing in large water bodies.

8.5.2 While the abundance of these radium isotopes – on an absolute basis and relative to each other – can provide useful information the concentration of these radium isotopes in large water bodies, especially in seawater, can be quite low and presents a challenge in measurement with needed precision. The geometry of MnO_2 impregnated on polymer fibers/cartridges provides a high surface area that when contacted with a large volume of water (tens, several hundred, even

several thousand litres) allows a representative sampling of the contained radium in a convenient manner (14-25). This contact with the MnO₂ impregnated polymer fibers/cartridges can be accomplished in-situ or using a collected and contained large volume water sample. Controlled studies have shown that the radium extraction efficiency is high but may not be 100 % so where an absolute radium isotope concentration is needed a grab sample of sufficient volume may need to be taken for fixed laboratory analysis. However, such grab sample radium isotope results may be combined with relative radium isotopic results to provide an overall useful set of radium isotope data.

8.5.3 The typical manner of impregnating the fiber/cartridge media with MnO₂ is to immerse in a similar volume of 0.5 M potassium permanganate solution at 70 to 80°C for 10 minutes. The reaction is exothermic and after the few minute contact the impregnated material should be transferred to deionized water for washing after which it may be dried and stored pending use.

8.5.4 After contact with the water being characterized for radium isotope content there are a variety of manners in which the radium content can be measured. These include gamma spectrometry of the dried fiber/cartridge in a standard geometry, measurement of the radon decay progeny from air

circulated through the dried fiber/cartridge, or elution of the radium from the fiber/cartridge media.

8.6 Use of MnO₂ Impregnated Thin Film:

8.6.1 While the approaches in the prior two sections made use of the associated high surface area of the substrate to maximize the advantage of the impregnated MnO₂ such a substrate geometry is not necessary where the aqueous sample volume is reasonably small and a longer contact time can be used. In this situation a thin polymer disc substrate geometry that can then be used for alpha spectrometry counting has notable value (26-29).

8.6.2 The thickness of the deposited MnO₂ can be on the order of 1 micrometer which still allows good alpha energy resolution to be obtained. The thickness of the deposited MnO₂ can be controlled through the deposition time but there may be a tradeoff with analyte recovery. The cited published work on this approach uses aqueous sample volumes of about 1 L and a contact time of up to 2 days.

9. Keywords

9.1 chemical separation; concentration; hydrous manganese oxide; manganese oxide; pre-concentration

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