TECHNICAL WHITE PAPER

Non-Radioactive Cyclohexane Method vs. Radioactive Methyl Iodide Method:

A Comparative Evaluation of Nuclear-Grade Activated Carbon Adsorber Test Methods

DIN 3754 | ANSI N509 / N510 | ASTM D3803 | NRC Regulatory Guide 1.52

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Abstract

Nuclear-grade activated carbon adsorbers are the primary engineered barrier against the release of radioactive organic iodine species, principally methyl iodide labeled with iodine-131 (CH3-131I), from nuclear power plant ventilation systems, hospital nuclear-medicine exhaust, radiochemical laboratory exhaust, and emergency air-cleaning systems. Two test methods dominate current industry practice for qualifying and surveilling this carbon: the non-radioactive cyclohexane method defined in DIN 3754, and the methyl iodide method defined in ANSI/ASME N509, ANSI N510, and ASTM D3803, and referenced by U.S. Nuclear Regulatory Commission Regulatory Guide 1.52. This white paper presents a systematic, side-by-side technical comparison of the two methods, including test conditions, underlying adsorption chemistry, regulatory acceptance, and the specific failure modes each method can and cannot detect. We conclude that the two methods are complementary rather than interchangeable: cyclohexane testing offers a fast, non-radioactive, low-cost screening tool suited to routine field surveillance, while methyl iodide testing remains the only method that directly evaluates the triethylenediamine (TEDA) chemisorption chemistry responsible for actual radioiodine capture, and is therefore required for regulatory qualification in the United States and recommended wherever a specific, guaranteed iodine removal efficiency is required.

Keywords: nuclear-grade activated carbon, cyclohexane method, DIN 3754, methyl iodide, ANSI N509, ANSI N510, ASTM D3803, TEDA, radioiodine, NRC Regulatory Guide 1.52, carbon adsorber qualification testing.

1. Introduction and Background

Nuclear-grade activated carbon adsorbers are the primary containment technology for radioactive iodine species — particularly iodine-131 (131I) and iodine-133 (133I) — in nuclear power plant ventilation, hospital nuclear-medicine exhaust, radiochemical laboratory exhaust, and emergency-response air-cleaning systems. A carbon adsorber that fails to capture organic radioiodine represents a direct release pathway to the environment and to occupied spaces; qualification testing of this carbon is therefore a regulatory requirement rather than a discretionary quality-control step.

Two fundamentally different testing approaches have evolved internationally to evaluate carbon filter performance. The cyclohexane method uses an organic vapor surrogate, cyclohexane (C_6H_{12}), to assess the general physical adsorptive capacity of the carbon without requiring the handling of radioactive or highly toxic materials; it is used primarily in Germany, in wider European practice, and for routine in-service surveillance. The methyl iodide method uses methyl iodide (iodomethane, CH_3I) — which may be 131I-labeled or non-radioactive — to directly test the carbon's capture of organic iodine species, representing the actual chemistry of radioiodine encountered in nuclear facilities; it is required by the U.S. Nuclear Regulatory Commission for NRC-licensed nuclear power plant applications.

The distinction between these two methods is not merely technical; it is regulatory. For NRC-licensed nuclear facilities in the United States, methyl iodide testing performed in accordance with ANSI N510 is required. In European practice, cyclohexane testing per DIN 3754 may be accepted for routine surveillance, but methyl iodide testing is typically still required for initial carbon qualification. This paper sets out the technical basis for that regulatory distinction.

2. The Cyclohexane Method (DIN 3754)

2.1 Method Overview

The DIN 3754 cyclohexane test method was developed in Germany as a practical, non-radioactive approach to the routine surveillance testing of activated carbon adsorbers installed in nuclear ventilation systems. By using cyclohexane (C_6H_{12}), a saturated alicyclic hydrocarbon, as a surrogate for organic radioiodine species, the method avoids the need for radioactive-material handling licenses and specialized containment facilities, making it well suited to field use by facility maintenance staff.

2.2 Test Conditions

Table 1 summarizes the standard test conditions specified under DIN 3754.

Table 1. DIN 3754 cyclohexane test conditions.

Parameter	Value / Specification
Test gas	Cyclohexane (C6H12) in air
Challenge concentration	~1,000 ppm C6H12 (approx. 3,400 mg/m3)
Temperature	20 C +/- 2 C
Relative humidity	50% +/- 5% RH
Face velocity	0.25 cm/s (typical surveillance velocity)
Result parameter	Dynamic Adsorption Capacity (DAC), mg C6H12 per gram carbon
New carbon acceptance	DAC >= 100 mg/g
In-service carbon acceptance	DAC >= 80 mg/g (typical)
Detection method	Flame ionization detector (FID) or gas chromatography (GC)
Primary standard	DIN 3754 (Germany); referenced in related ISO/EN documents

2.3 Advantages

- No radioactive material handling: no license required, no contamination risk, no radioactive waste generated.
- Lower-hazard chemistry: cyclohexane is substantially less hazardous than methyl iodide, which is classified as a carcinogen and an alkylating agent.
- Speed and cost: the test can typically be completed in one to two hours using simpler, less expensive equipment than a methyl iodide test setup.
- Field-deployable: suitable for routine in-service surveillance performed directly by nuclear facility maintenance staff without specialized radiological controls.
- Established practice: decades of operational data exist for DIN 3754 cyclohexane testing in European nuclear facilities.

2.4 Limitations

- Not representative of radioiodine chemistry: cyclohexane is captured primarily by physisorption (van der Waals forces), whereas methyl iodide is captured by chemisorption through reaction with the TEDA impregnant. The two mechanisms follow different kinetics.
- Not accepted by the NRC: the U.S. Nuclear Regulatory Commission does not accept cyclohexane testing as a substitute for methyl iodide testing in nuclear power plant applications.
- Cannot assess TEDA reactivity: the cyclohexane test does not evaluate TEDA impregnation level or its chemical reactivity with methyl iodide, which is the single most critical performance parameter for radioiodine capture.
- Different humidity dependence: carbon capacity for cyclohexane varies with humidity differently than for methyl iodide, which limits the validity of cross-method comparisons.

3. The Methyl Iodide Method (ANSI N509/N510, ASTM D3803)

3.1 Method Overview

The methyl iodide (CH3I) test is the current industry and regulatory standard for nuclear-grade activated carbon adsorber qualification. It uses methyl iodide as a direct chemical surrogate for organic radioiodine species — principally CH3-131I — which represent the most penetrating and difficult-to-capture form of radioiodine present in nuclear facility air streams. Unlike the cyclohexane method, the methyl iodide test directly challenges the carbon's iodine capture chemistry, encompassing both physisorption and the TEDA-mediated chemisorption reaction, under conditions representative of actual nuclear facility operation.

While elemental iodine (I2) is also present in nuclear facility air streams, organic iodides — particularly methyl iodide — are the most challenging species to capture on activated carbon, because CH3I is less reactive with TEDA than elemental I2 and penetrates more readily through improperly impregnated or aged carbon. Carbon that passes the methyl iodide test will also adequately capture elemental I2; the converse is not necessarily true. This asymmetry is the technical basis for using CH3I as the regulatory challenge species.

3.2 Test Conditions

Table 2. Methyl iodide test conditions per ANSI N509/N510, ASTM D3803, and NRC Regulatory Guide 1.52 Revision 4.

Parameter	Value / Specification
Test gas	Methyl iodide (CH3I / CH3-131I) in air
Challenge concentration	10 mg CH3I per m3 air (approx. 1.7 ppm)
Temperature	25-30 C (NRC: 30 C +/- 2 C per RG 1.52)
Relative humidity	70% +/- 5% RH (worst-case for TEDA performance)
Face velocity	0.5 cm/s (maximum rated face velocity)
Bed depth	Per actual installed depth (minimum 2 inches / 51 mm typical)
Exposure duration	2 hours minimum (ASTM D3803)
Result parameter	CH3I penetration (%) = downstream / upstream concentration x 100
Alternative expression	Decontamination Factor (DF) = 1 / penetration fraction
NRC acceptance criterion	<= 0.175% CH3I penetration (DF >= 571) per NRC RG 1.52 Rev. 4
Detection method	131I gamma counting (radioactive CH3I) or gas chromatography (non-radioactive CH3I)
Primary standards	ANSI/ASME N509, ANSI N510, ASTM D3803, NRC RG 1.52

3.3 Significance of the 70% Relative Humidity Condition

The 70% relative humidity test condition is deliberately conservative. Water vapor competes with methyl iodide for TEDA active sites on the carbon surface, reducing effective capacity. Carbon that performs adequately at 70% RH is expected to perform even better under the lower-humidity conditions typical of actual nuclear facility air systems. Specifying 70% RH as the standard test condition therefore provides an engineering safety margin while remaining an achievable target for properly impregnated TEDA carbon.

4. Comparative Analysis

Table 3 presents a direct, parameter-by-parameter comparison of the two test methods.

Table 3. Side-by-side comparison of the cyclohexane and methyl iodide test methods.

Parameter	Cyclohexane Method (DIN 3754)	Methyl Iodide Method (ANSI N509/N510, ASTM D3803)
Test gas	Cyclohexane, C6H12	Methyl iodide, CH3I
Radioactive material required	No	Optional (131I-labeled or non-radioactive CH3I)
Challenge concentration	~1,000 ppm (~3,400 mg/m3)	10 mg/m3 (~1.7 ppm)
Test temperature	20 C +/- 2 C	25-30 C (NRC: 30 C +/- 2 C)
Relative humidity	50% +/- 5%	70% +/- 5% (worst-case)
Face velocity	0.25 cm/s	0.5 cm/s
Result expression	Dynamic Adsorption Capacity, DAC (mg/g)	CH3I penetration (%) or Decontamination Factor (DF)
Acceptance criterion (new carbon)	DAC >= 100 mg/g	<= 0.175% penetration (DF >= 571)
Acceptance criterion (in-service carbon)	DAC >= 80 mg/g	<= 0.175% penetration (DF >= 571)
Detects TEDA reactivity directly	No	Yes
Representative of actual 131I chemistry	Surrogate only	Direct chemical analog
NRC-accepted for U.S. nuclear power plants	Not accepted	Required
Typical use	Routine in-service surveillance (Europe)	Initial qualification and regulatory compliance (U.S. and international)

The comparison in Table 3 makes clear that the two methods are not interchangeable substitutes but serve different purposes within a carbon qualification program: the cyclohexane method is a rapid, low-hazard screening and surveillance tool, while the methyl iodide method is the only method that directly and quantitatively verifies the chemisorption performance responsible for actual radioiodine removal.

5. TEDA Impregnation Chemistry

Unimpregnated (virgin) activated carbon, regardless of its pore structure or surface area, has insufficient performance for nuclear-grade methyl iodide capture; penetration through virgin carbon can be many orders of magnitude above the NRC acceptance criterion of 0.175%. Impregnation of the carbon with triethylenediamine (TEDA, also known as DABCO, 1,4-diazabicyclo[2.2.2]octane) is therefore essential to nuclear-grade performance.

TEDA reacts with methyl iodide through a nucleophilic substitution (SN2) reaction. The nitrogen lone pair of TEDA attacks the methyl carbon of CH3I, displacing the iodide ion and forming a stable quaternary ammonium salt: TEDA + CH3I -> [TEDA-CH3]+ I-. This chemisorption reaction is effectively irreversible under normal operating conditions, which explains both the high efficiency and the long-term stability of properly impregnated TEDA carbon in service.

Representative TEDA carbon specification parameters used in nuclear-grade adsorber manufacturing include: TEDA content typically 2-5% by weight (the exact formulation is proprietary to each manufacturer); BET surface area of at least 1,000 m2/g for coconut-shell-based nuclear carbon; ball-pan hardness of at least 90%, to resist attrition in service; as-received moisture content of no more than 5%; ash content of no more than 5%; and particle size typically 6x16 mesh (3.35 x 1.19 mm) for nuclear applications.

6. NRC Regulatory Framework

U.S. Nuclear Regulatory Commission Regulatory Guide 1.52, Revision 4 (2012), establishes design, testing, and maintenance criteria for nuclear power plant air-cleaning systems. Table 4 summarizes the key provisions applicable to activated carbon adsorbers.

Table 4. Key NRC Regulatory Guide 1.52 (Rev. 4) provisions for carbon adsorbers.

Requirement	NRC Specification (Regulatory Guide 1.52, Revision 4)
Test method	ASTM D3803 / ANSI N510 methyl iodide test
CH3I concentration	10 mg/m3
Temperature	30 C +/- 2 C
Relative humidity	70% +/- 5% RH
Face velocity	<= 0.5 cm/s
Acceptance criterion	<= 0.175% CH3I penetration (DF >= 571)
Initial carbon qualification	Tested per ASTM D3803 prior to installation
In-service surveillance	Representative carbon samples tested per ANSI N510 at defined intervals
Cyclohexane test (DIN 3754)	Not accepted as a substitute for U.S. nuclear power plant applications

Thirty-seven U.S. states hold Agreement State status and operate their own nuclear regulatory programs. While NRC Regulatory Guide 1.52 applies specifically to nuclear power plants, Agreement State programs govern medical, research, and industrial nuclear users and may impose different or additional requirements; facility operators should verify current requirements with their applicable state radiation control program. As a general principle, wherever radioiodine capture efficiency has direct public-health significance, methyl iodide testing provides the only direct, quantitative measurement of actual carbon performance.

7. Method Selection Guidance

7.1 When to Use Methyl Iodide (CH3I) Testing

- NRC-licensed nuclear power plant or fuel-cycle facility applications (required).
- Initial qualification of a new carbon lot for nuclear service.
- Regulatory compliance documentation.
- High-dose hospital iodine-131 therapy room exhaust filtration.
- Radiochemical laboratory or hot-cell ventilation.
- Any application in which a specific, guaranteed iodine removal efficiency must be demonstrated.

7.2 When Cyclohexane (DIN 3754) Testing Is Appropriate

- Routine in-service surveillance at European nuclear facilities, as a supplement to initial CH3I qualification.
- Rapid field assessment of carbon condition without radioactive-material handling.
- Industrial, non-nuclear activated-carbon capacity screening.
- Research comparison of carbon lots where only relative capacity is required.
- Pre-qualification screening, to identify obviously depleted carbon before committing to methyl iodide testing.

7.3 Critical Caution

Cyclohexane testing should not be substituted for methyl iodide testing in any application where regulatory compliance with NRC requirements, ANSI N509/N510, or ASTM D3803 is required. A carbon bed that passes DIN 3754 cyclohexane testing may nonetheless fail to meet ANSI N510 methyl iodide requirements — particularly if the TEDA impregnant has degraded, leached, or been exposed to acid gases, solvent vapors, or excessive humidity. Acid gases such as hydrogen chloride (HCl) and sulfur dioxide (SO2) react preferentially with TEDA, destroying its nucleophilic reactivity and reducing CH3I capture efficiency, while leaving cyclohexane physisorption capacity comparatively unaffected. This is why annual (or event-triggered) methyl iodide testing of in-service nuclear carbon is considered critical and cannot be replaced by cyclohexane surveillance alone.

8. Conclusion

The cyclohexane method (DIN 3754) and the methyl iodide method (ANSI N509/N510, ASTM D3803) address different needs within a nuclear-grade carbon adsorber qualification program and should be understood as complementary rather than competing approaches. Cyclohexane testing offers a fast, low-hazard, field-deployable means of routine surveillance, but it cannot evaluate the TEDA chemisorption chemistry that governs actual radioiodine capture, and is therefore not accepted by the NRC for U.S. nuclear power plant applications. Methyl iodide testing, while requiring greater analytical infrastructure and, in some configurations, radioactive-material handling, remains the only method that directly and quantitatively verifies the chemistry responsible for organic radioiodine capture, and is required wherever regulatory compliance or a guaranteed removal efficiency is at stake. A well-designed carbon qualification and surveillance program uses methyl iodide testing for initial qualification and regulatory compliance, and may supplement it with cyclohexane or comparable non-radioactive screening methods for routine field monitoring between full qualification cycles.

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About Iodine Air Systems

Iodine Air Systems, a brand of SortLease LLC (Sheridan, Wyoming, USA), designs and manufactures nuclear-grade air purification and carbon adsorber systems for nuclear power, hospital nuclear-medicine, radiochemical laboratory, and emergency-response applications. IAS carbon adsorber products use nuclear-grade TEDA-impregnated activated carbon, factory-qualified by methyl iodide testing in accordance with ANSI N509/N510 prior to shipment. Each carbon filter element ships with a factory CH_3I penetration test certificate, TEDA content verification, physical properties data (BET surface area, hardness, particle size, moisture, and ash content), and batch traceability documentation.

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