ISSN: 0975-833X

Available online at http://www.journalcra.com

International Journal of Current Research
Vol. 18, Issue, 02, pp.36262-36267, February, 2026
DOI: https://doi.org/10.24941/ijcr.50153.02.2026

INTERNATIONAL JOURNAL OF CURRENT RESEARCH

RESEARCH ARTICLE

FORENSIC WORKFLOW FOR RESIDUE RECOVERY FROM OVERSIZED POST-BLAST EXHIBITS IN ANFO DETONATIONS

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ARTICLE INFO

Article History:
Received 25th November, 2025
Received in revised form
20th December, 2025
Accepted 18th January, 2026
Published online 27th February, 2026

Keywords:
Post-blast Analysis; Oversized Exhibits; ANFO; GC–MS; FTIR; Forensic Workflow.

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ABSTRACT

Post- blast investigations involving ammonium nitrate fuel oil (ANFO) often generate oversized debris with irregular surfaces, complex matrices, and uneven residue distribution, limiting the effectiveness of conventional forensic recovery methods. This study proposes a workflow-centred forensic approach specifically designed for residue recovery from oversized post-blast exhibits following ANFO detonations. The workflow emphasises structured surface coverage through spatial subsampling, sequential solvent swabbing, and syringe filtration to mitigate heterogeneity and matrix-derived interference prior to instrumental analysis. Organic and inorganic residues recovered through this process were examined using thin-layer chromatography (TLC), gas chromatography–mass spectrometry (GC–MS), classical chemical tests, and Fourier-transform infrared (FTIR) spectroscopy. The analytical results served primarily to evaluate workflow performance rather than to introduce new chemical signatures. Diesel-range hydrocarbons and nitrate-based oxidisers, including ammonium and potassium nitrate, were consistently identified, while chlorates, perchlorates, and metallic additives were absent. Spatially informed sampling improved representative residue collection across large surfaces, and syringe filtration enhanced extract cleanliness and analytical stability. The study demonstrates that adapting forensic procedures to the scale and heterogeneity of post-blast exhibits is critical for reliable residue recovery. The proposed workflow offers a reproducible and casework-oriented framework for forensic laboratories handling oversized debris in ANFO-related explosive investigations.

Citation: Kumar D, Niranjan Baliarsingh and Soujanya Gowneni. 2026. "Forensic Workflow for Residue Recovery from Oversized Post-Blast Exhibits in ANFO Detonations.". ***International Journal of Current Research, 18, (02), 36262-36267.***

INTRODUCTION

The forensic investigation of explosive incidents plays a critical role in event reconstruction, identification of explosive formulations, and judicial proceedings. Ammonium nitrate fuel oil (ANFO) remains one of the most frequently encountered improvised explosives due to its widespread availability, low cost, and high detonation efficiency (1,2). Following detonation, explosive residues are dispersed onto a wide range of substrates, including soil, metals, plastics, and concrete. Oversized and irregular debris presents additional analytical challenges because residue deposition is often uneven, resulting in pronounced surface contamination gradients (3). Conventional residue recovery techniques, such as swabbing and solvent rinsing, are routinely applied in forensic casework. However, these methods are primarily optimized for small and relatively uniform exhibits and often yield poor recovery when applied directly to large, heterogeneous post-blast debris (4). To address these limitations, adaptations such as spatially resolved subsampling, improved filtration, and optimized solvent extraction strategies have been proposed (Figure 1) (5).

Analytical techniques remain central to post-blast residue characterization. Gas chromatography–mass spectrometry (GC–MS) provides reliable detection of organic explosive components and fuel oils, while Fourier-transform infrared (FTIR) spectroscopy and classical chemical spot tests are widely used for the identification of inorganic oxidizers and ions (6,7). The effectiveness of these techniques, however, is strongly dependent on the efficiency of the initial sampling and extraction procedures.

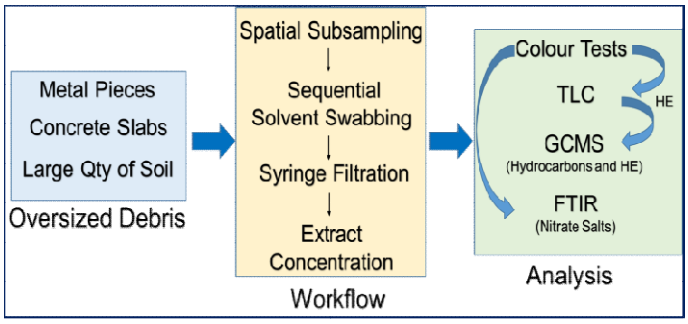

Figure 1. Forensic Workflow of Oversized Exhibits Examination

While recent advances in chemosensors and molecular frameworks have demonstrated high selectivity for nitroaromatic explosives under controlled conditions (12,13), such approaches are generally unsuitable for complex and contaminated post-blast debris. Comprehensive reviews, including the INTERPOL survey on explosives analysis, highlight the lack of validated workflows for heterogeneous and oversized exhibits and emphasize contamination control as a critical factor in forensic reliability (14). The present study addresses this gap by developing and validating an integrated forensic workflow—combining sequential swabbing, solvent extraction, syringe filtration, and spatial subsampling with complementary analytical techniques—specifically tailored for oversized ANFO-related post-blast exhibits.

MATERIALS AND METHODS

Exhibits Received for Examination: Oversized metallic, plastic, and soil fragments were received for forensic examination from the investigating authority following a post-blast incident involving an ANFO detonation. The exhibits comprised large metal fragments measuring up to 29 inches in length, spades, metallic pots approximately 12 inches in height, a cardboard drum, and a deformed metallic cover (Figure 2). Control soil samples were collected from unaffected areas in the vicinity of the blast site to serve as negative controls. All exhibits and control samples were packaged individually in clean, sealed containers to prevent cross-contamination during handling and transport. (16–18)

Figure 2. Oversized Exhibits collected from the Crime Scene

Residue Recovery and Spatial Subsampling Strategy: Residues were recovered from oversized post-blast exhibits using a structured residue recovery approach based on sequential swabbing. Cotton applicators were moistened with analytical-grade solvents and applied in a defined sequence—diethyl ether, acetone, deionized water, sodium hydroxide solution, and pyridine—to preferentially extract non-polar organic residues followed by polar organic and inorganic components. Swabbing was performed systematically across all accessible surfaces of each exhibit to ensure comprehensive coverage. The collected swabs were extracted into clean glassware, and the combined extracts were filtered through 0.22 µm nylon syringe filters to remove particulate matter and minimize matrix-related interference. The filtrates were subsequently concentrated at room temperature to a final volume of approximately 2–5 mL prior to instrumental analysis. (10,11,16–18). Oversized post-blast exhibits frequently display highly heterogeneous residue distribution as a result of uneven deposition, secondary fragmentation, and surface-specific adsorption processes. To address this variability, a spatial subsampling strategy was implemented in which large exhibits were systematically divided into discrete surface regions based on geometry, exposure orientation, and visible blast effects.

Each region was independently swabbed and extracted rather than combined into a single composite sample, thereby reducing dilution of localized residue hotspots and increasing the probability of recovering trace-level explosive components from contaminated or weathered surfaces (Figure 3). In addition, spatial subsampling enabled comparative assessment of residue distribution across different areas of the same exhibit, providing valuable contextual information for forensic interpretation. When integrated with sequential swabbing and syringe filtration, this approach significantly enhanced analytical sensitivity, reproducibility, and interpretative reliability for heterogeneous and oversized post-blast debris associated with ANFO detonations.

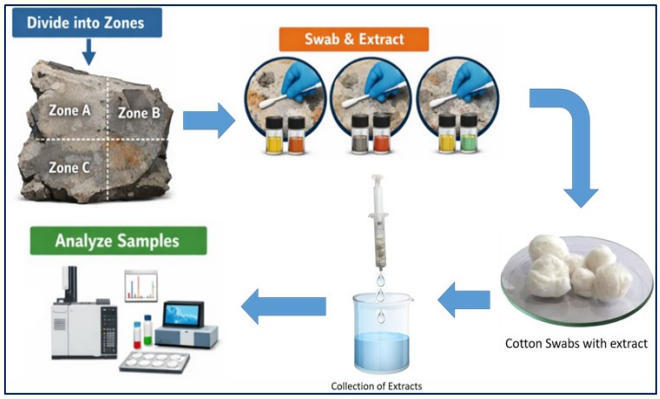

Figure 3. Spatial subsampling strategy

Analytical Workflow: A schematic overview of the integrated analytical workflow is shown in Figure 4 based on the standard laboratory protocols. (9)

Organic residue analysis: Ether and acetone extracts were examined using thin-layer chromatography (TLC) on silica gel plates, employing chloroform/acetone (1:1, v/v) and toluene/cyclohexane (7:3, v/v) as mobile phases. Visualization was carried out under UV light (254 nm) and by chemical spraying. Confirmatory analysis was performed using GC–MS for the detection of fuel-oil hydrocarbons and high explosives. (8–11)

Inorganic residue analysis: Water and sodium hydroxide extracts were subjected to classical chemical spot tests for common anions and cations, including nitrate, nitrite, ammonium, and potassium. Selected extracts were further characterized using FTIR spectroscopy.

Filtration assessment: The effect of syringe filtration on analytical clarity and background interference was evaluated by comparing filtered and unfiltered extracts during GC–MS analysis. (16–18).

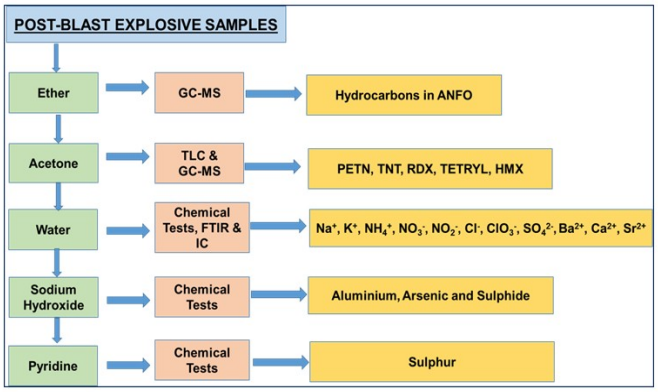

Figure 4. Schematic diagram for Post-Blast Explosive Analysis

Instrumental Parameters

- **GC–MS**: Analyses were carried out with an electron ionization (EI) source at 70 eV, full-scan mode (m/z 40–500), on a 30 m × 0.25 mm × 0.25 µm capillary column. The oven program ramped from 100 °C to 280 °C with controlled heating rates. Compounds were identified by comparison with NIST library spectra. Table 1 briefs the parameters of the GC-MS method. (8-11)
- **TLC**: Developed plates were visualized under UV (254 nm) and by spraying with diphenylamine and sulfuric acid reagents. (8-11)
- **FTIR**: ATR-FTIR spectra were recorded from 4000–400 cm^{-1} at 4 cm^{-1} resolution; spectral matching was performed using instrument libraries. (8-11)

Table 1. Parameters of the GC-MS Method

MS transfer line temperature [°C]	250
Ion source temperature [°C]	250
Total Run time [min]	6.250
Inlet Temperature [°C]	230
Carrier Flow [ml/min]	1.000
GC Oven Temperature Nominal	Start at 100.0 °C hold for 0.2 min
	Ramp to 220 °C at 30 °C/min
	Hold for 0.3 min at 220 °C
	Ramp to 280 °C at 80°C/min
	Hold for 1 min at 280°C
°C - degree Celsius, min-minutes	

Quality Control: Procedural blanks (blank swabs) and negative control samples (control soil) were processed concurrently with questioned exhibits to assess potential environmental and laboratory-derived contamination throughout all stages of the analytical process. Certified reference materials and laboratory standards of commonly encountered explosives and explosive components, including RDX, TNT, PETN, and ANFO constituents, were analyzed under identical instrumental and procedural conditions to support analytical verification and result interpretation. The inclusion of negative controls and reference materials as part of routine quality assurance and quality control measures is consistent with INTERPOL-recommended best practices and ASTM guidance for the forensic examination of explosives and explosive residues, which emphasize contamination monitoring, method validation, and interpretive reliability as essential elements of forensic reporting. (14)

Challenges in the Analysis of Oversized Exhibits: Extraction from the exhibits of smaller sizes and their debris is possible by rinsing the exhibit with a minimum quantity of solvent in a beaker. In this method, the ingredients of the unexploded and exploded explosives can be easily collected from the exhibits by dissolution. In contrast, oversized exhibits cannot be conveniently immersed or rinsed, making swabbing the primary viable recovery method. Factors such as cotton quality, operator variability, solvent loss, and inefficient transfer of residues from the swab can affect recovery efficiency. Careful and systematic swabbing, combined with filtration and complementary analytical techniques, is therefore essential to minimize analytical uncertainty.

Observations

TLC and GC-MS Examinations: GC–MS analysis of ether extracts revealed the presence of high-boiling petroleum hydrocarbons. Hexadecane was identified at a retention time of 14.02 min, with a similarity index (SI) of 792 and a reverse similarity index (RSI) of 929 based on NIST library matching. The corresponding total ion chromatogram (TIC) is shown in Figure 5. No high explosives were detected in acetone extracts by TLC or GC–MS. (16-18)

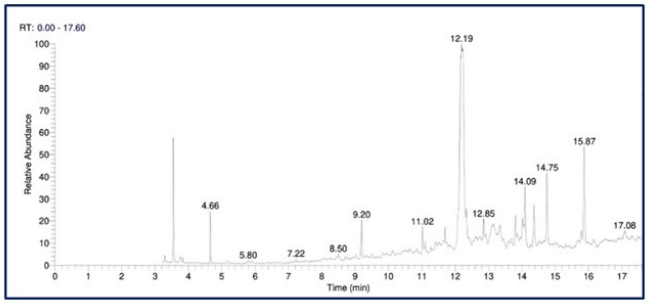

Figure 5. TIC of the exhibit having hydrocarbons

The mass spectra of the forensic case sample obtained by analysis using the GC-MS method, along with the reference library of mass spectra, are shown in Figure 6. The x axis presents abundance and y axis presents m/z values. (16-18)

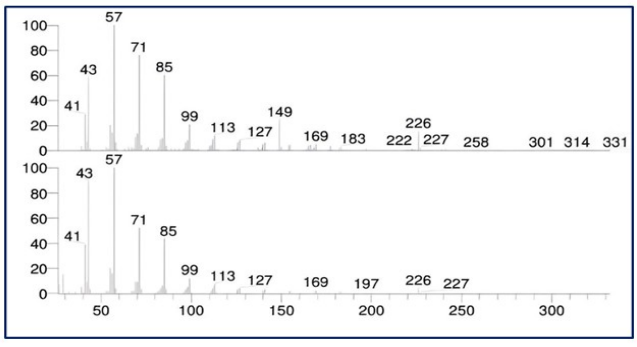

Figure 6. Mass spectrum of the Extract (Top) and its NIST Library (Bottom)

Chemical Examinations: Chemical examination of water, alkaline, and pyridine extracts yielded positive results for nitrate, nitrite, ammonium, potassium, chloride, sulfate, and elemental sulfur in the questioned exhibits. Chlorate, perchlorate, and metallic additives such as aluminium and magnesium were not detected. Observations are summarized in Table 2. (16-18)

Table 2. Observations of the Chemical Examinations

Sl.No	Chemical Test	Target Ion/Analyte	Observation
1	Silver Nitrate	Chloride	Present
2	Griess Test	Nitrite	Present
3	Griess reagent + Zn dust	Nitrate	Present
4	Aniline sulphate	Chlorate	Absent
5	Methylene blue indicator	Perchlorate	Absent
6	Barium chloride	Sulphate	Present
7	Zinc Uranyl Acetate	Sodium	Absent
8	Sodium Cobaltinitrate	Potassium	Present
9	Nessler's reagent	Ammonium	Present
10	Magneson-I	Magnesium	Absent
11	Sodium Rhodizonate	Barium, Calcium, Strontium	Absent
12	Sodium Nitroprusside	Sulphide (NaOH Extract)	Absent
13	Alizarine-S	Metallic Aluminium (NaOH Extract)	Absent
14	Gutzeit's Test	Arsenic (NaOH Extract)	Absent
15	Pyridine + NaOH	Elemental Sulphur	Present

FTIR Analysis: FTIR analysis of aqueous extracts confirmed the presence of nitrate-based oxidizers. Reference spectra of ammonium nitrate and potassium nitrate were used for comparison. Both oxidizers were detected across different extracts, indicating heterogeneous residue distribution on oversized exhibits. Representative spectra are shown in Figures 7 and 8. (16-18)

RESULTS

Organic Residue Detection (GC–MS and TLC): GC–MS analysis of ether extracts confirmed the presence of high-boiling petroleum hydrocarbons, with hexadecane detected at a retention time of 14.02 min (Figure 5). This compound is characteristic of diesel oil fractions and supports the presence of fuel oil components consistent with ANFO formulations. The mass spectral profile exhibited a high similarity match with the NIST library, indicating reliable compound identification (Figure 6). No high-explosive compounds were detected in acetone extracts by either TLC or GC–MS analysis, suggesting the absence of secondary high-explosive admixtures within the recovered residues.

Inorganic Residue Detection by Chemical Tests: Chemical spot tests yielded positive responses for nitrite, nitrate, ammonium, chloride, potassium, sulphate ions, and elemental sulphur across multiple post-blast exhibits (Table 2). Tests for chlorate, perchlorate, and metallic additives such as aluminium and magnesium were negative. These results are consistent with nitrate-based low explosive residues typically associated with ANFO formulations.

FTIR Characterization of Post-Blast Inorganic Oxidizer Residues: FTIR spectroscopy was employed to characterize inorganic oxidizer residues recovered from oversized post-blast exhibits. Spectra obtained from questioned samples were compared with laboratory reference spectra of ammonium nitrate and potassium nitrate. This variation reflects spatial heterogeneity, with ammonium nitrate detected in some subsamples and potassium nitrate predominating in others

The ammonium nitrate reference spectrum exhibited a broad N–H stretching band at approximately 3392 cm^{-1}, along with characteristic absorptions at ~1636 cm^{-1} (ammonium bending) and a strong nitrate asymmetric stretching band at ~1322 cm^{-1} . Additional nitrate-related absorptions were observed at ~1045 cm^{-1} and ~820 cm^{-1}, with lattice vibration bands below 600 cm^{-1}. In contrast, the questioned post-blast samples lacked the ammonium N–H stretching feature near 3400 cm^{-1} and displayed a dominant absorption at ~1364 cm^{-1}, characteristic of nitrate asymmetric stretching in alkali metal nitrates. Additional absorptions at ~953 cm^{-1}, ~823 cm^{-1}, and multiple bands within the 730–560 cm^{-1} region were consistent with potassium nitrate. The absence of ammonium-specific bands supported exclusion of ammonium nitrate as the primary oxidizer in these subsamples.

Workflow Performance in Oversized Exhibits: Sequential swabbing followed by solvent extraction and syringe filtration resulted in improved extract clarity and enhanced analytical performance. Syringe filtration effectively reduced particulate interference and background noise during GC–MS analysis, as reflected by improved chromatographic resolution (Figure 6), facilitating reliable detection of trace-level residues (4). Spatially resolved subsampling (Figure 3) further contributed to improved recovery from heterogeneous surfaces, minimizing dilution of localized residue hotspots.

DISCUSSION

Forensic Interpretation of Analytical Findings: The combined recovery of petroleum hydrocarbons and nitrate salts confirms the ANFO origin of the post-blast residues and is consistent with molecular fingerprints reported for ANFO detonations in technical reference studies (15). The identification of diesel-range hydrocarbons by GC–MS (Figure 6), together with nitrate-based inorganic residues detected by chemical tests (Table 2) and FTIR spectroscopy (Figure 8), provides complementary and mutually reinforcing evidence supporting device composition and formulation. Compared with prior studies employing molecular frameworks and chemosensors for selective detection of nitroaromatic explosives (12,13), the present workflow directly addresses the analytical challenges posed by large-scale, heterogeneous, and environmentally contaminated post-blast debris. While sensor-based approaches demonstrate high selectivity under controlled laboratory conditions, their applicability to mixed and contaminated post-blast matrices remains limited. In contrast, the integrated methodology described here—incorporating spatial subsampling (Figure 3), sequential solvent swabbing, syringe filtration, and complementary analytical techniques—provides a robust and adaptable framework for forensic examination of oversized exhibits. FTIR spectroscopy proved particularly valuable as a rapid screening tool for differentiating between ammonium-based oxidizers and alkali metal nitrates (Figure 7 and 8), thereby guiding subsequent confirmatory analyses and contextual interpretation. Overall, the workflow enhances analytical sensitivity, improves reproducibility, and strengthens evidentiary reliability, extending forensic capability in the investigation of complex ANFO-related detonation scenarios involving oversized post-blast debris.

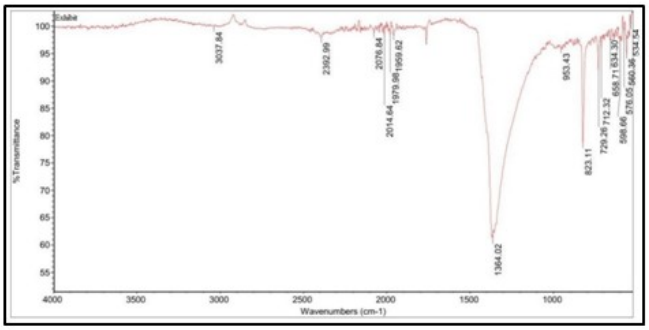

Figure 7. FTIR spectrum of Potassium Nitrate

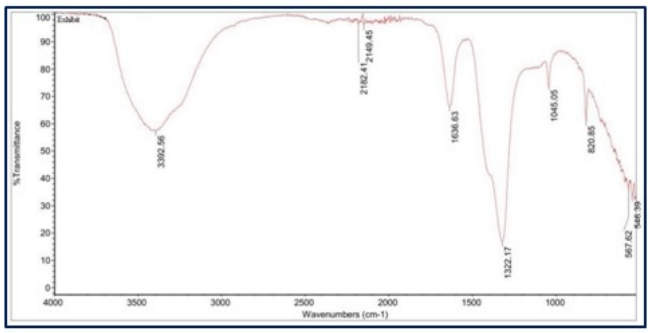

Figure 8. FTIR spectrum of Ammonium Nitrate

CONCLUSION

This study demonstrates that tailored forensic workflows are essential for the reliable analysis of oversized post-blast exhibits. By integrating sequential swabbing, solvent extraction, syringe filtration, and spatial subsampling with complementary analytical techniques, both organic (fuel oil hydrocarbons) and inorganic (nitrate-based) residues were detected with high confidence. Syringe filtration was particularly effective in maximizing recovery and reducing analytical interference, while multi-method corroboration strengthened evidentiary interpretation. These findings confirm the exclusive use of ANFO in the investigated detonation and highlight the broader significance of adapting forensic protocols to heterogeneous, large-scale evidence. Beyond the present case, the validated workflow provides a generalizable framework that enhances evidentiary reliability and strengthens the chemical basis for reconstructing complex detonation events.

Abbreviations

The following abbreviations are used in this manuscript:

TLC	Thin Layer Chromatography
GC-MS	Gas Chromatography Mass Spectrometry
FTIR	Fourier Transform Infrared Spectroscopy
in.	inches
SI	Similarity Index
RSI	Reverse Similarity Index
TIC	Total Ion Chromatogram
NIST	National Institute of Standards and Technology
AR	Analytical Reagent
ATR	Attenuated Total Reflectance
ANFO	Ammonium Nitrate Fuel Oil
PETN	PentaErythritol Tetra Nitrate
TNT	Tri Nitro Toluene
RDX	Research Department Explosive/ Royal Demolition Explosive
TETRYL	Trinitrophenylmethylnitramine
HMX	High Melting Explosive - Octogen
UV	Ultra Violet
NaOH	Sodium Hydroxide

Declarations

Ethics approval and consent to participate: Not applicable

Consent for publication: The authors have reviewed, revised, and approved the final manuscript for submission.

Funding: None

Clinical trial number: Not applicable

Author Contributions: Conceptualization, D.K.; methodology, D.K.; validation, D.K.; formal analysis, D.K.; investigation, D.K.; writing—original draft preparation, D.K; writing—review and editing, D.K, N.B., S.G.; visualization, D.K, N.B., S.G.; and supervision, D.K, N.B., S.G. The authors have read and agreed to the published version of this manuscript.

Institutional Review Board Statement: Not applicable.

Data Availability Statement: The authors declare that the data supporting the findings of this study are available within the paper. Should any raw data files be needed in another format, they are available from the author upon reasonable request.

Acknowledgments: We would like to express my gratitude to the Director, Central Forensic Science Laboratory, Pune and Hyderabad for continuous support and for providing the necessary infrastructure for this study.

Conflicts of Interest: The authors declare that there is no conflicts of interest.

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