

renal duplication progress to functional loss in one moiety, and early detection of such exclusion can prevent unnecessary interventions. ^{99m}Tc -DTPA scintigraphy also enables indirect identification of vesicoureteral reflux even without cystography, as in this case, where reflux of the tracer into the functionally excluded moiety supported the diagnosis and guided therapeutic decisions. **Conclusion:** This case demonstrates the importance of dynamic renal scintigraphy with ^{99m}Tc -DTPA in assessing renal function in complex urologic malformations. The identification of functional exclusion of the lower moiety and vesicoureteral reflux was fundamental in multidisciplinary management and treatment planning.

Keywords: Kidney Diseases, Congenital, Duplex Kidney, Vesico-Ureteral Reflux, Renal Scintigraphy, Technetium Tc 99m DTPA, Chronic Kidney Disease.

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CASE REPORTS

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VALIDATION OF THIN LAYER CHROMATOGRAPHY (TLC) FOR RADIOCHEMICAL IMPURITY DETERMINATION OF ^{68}Ga -PSMA-11

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European Pharmacopoeia (EP) published in 2024 the monograph “Gallium (^{68}Ga) PSMA-11 injection”, one of the most used PET-radiopharmaceutical for prostate cancer diagnosis, establishing the limit and procedure for radiochemical impurity determination by TLC. When the method for release the product is pharmacopendial, it is not required to validate the analytical method; instead, it must be performed the verification of the method to demonstrate the performance under the laboratory conditions. This work aimed to validate the TLC method used to quantify % free Ga-68 after labeling BLINDING PSMA-11 cold kit formulation, performing repeatability, intermediate precision, selectivity, linearity, and robustness tests. TLC was performed as described in EP. Radioactive samples of 37 MBq/mL were applied to iTLC strips (1.5×12.5 cm) at 1.5 cm from the base. Automatic gamma

counter (Cobra II, Packard) was used to measure the radioactivity of the strips at 300–1200 keV energy window. For assessment the repeatability, 6 samples were prepared by the same analyst. For intermediate precision, samples were tested by another analyst; both assays evaluated the relative standard deviation (RSD) of % free Ga-68. The selectivity evaluate the retention factor (Rf) and the profile of the chromatographic separation of 6 samples containing or not the following solutions 0.1 mL of ^{68}Ga -PSMA-11, $^{68}\text{GaCl}_3$ or excipient (0.02 mL of 100 mg/mL mannitol) in final volume of 1 mL. The analytical curve was obtained with radioactive concentrations solutions of 1.11, 3.7, 18.5, 29.6, 37, and 44.4 MBq/mL. The counting of the strips considered Ga-68 decay using the time of beginning of the test. Counting of the strips related to free Ga-68 should be proportional to concentration, the slope must be significantly different from zero, and the correlation coefficient must be above 0.990. Linearity analysis was performed using statistical methods. Robustness evaluated the influence of sample radioactive concentration (44 MBq/mL and 18.5 MBq/mL), sample volume (5 μL and 2 μL), and stationary phase (iTLC and TLC). The acceptance criteria established for this assay was $\text{Ga-68} < 3\%$, Rf Ga-68 of 0.2 and $\text{RSD} = 25.0\%$. All assays were performed in triplicate. The RSD of repeatability and intermediate precision assays were 9.36 and 12.4%, respectively. According to TLC profile, the Rf of ^{68}Ga -PSMA-11 was 0.8–1.0 and the selectivity test showed that there is no variation in the Rf of the product and free Ga-68 even in the presence of mannitol (component of the cold kit formulation). In linearity, Cochran test showed homoscedasticity of the results, demonstrating that the use of ordinary least squares (OLS) is adequate, F-test confirmed the linear correlation between radioactive concentration and counting, and the variance of the analytical curve. Shapiro-Wilk test demonstrated normal distribution of the residuals, and consequently, Grubbs test indicated that there are no outliers. Slope and correlation coefficient were 24438 and 0.997, respectively. Robustness assays yielded values up to RSD 14.22%, obtained with 0.5 mCi/mL, 2.0 μL sample volume, and iTLC strips. The TLC method described in EP 11th Edition to determine % free Ga-68 after the labeling PSMA-11 cold kit was evaluated in accordance to ANVISA Normatives (RDC 166/2017 and Guide 10 for statistical analyses, 2017), demonstrating selectivity, repeatability, intermediate precision, linearity, and robustness.

Keywords: Radiopharmaceutical, ^{68}Ga -PSMA-11, Validation, Thin-layer chromatography.

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