Synthesis of Glovebox Atmosphere Indicator (Atmoscheck)

In an oven-dried 250 mL round bottom schlenk flask, Cp_2TiCl_2 (2g, red crystals) and zinc powder (2+ grams, excess) were placed under nitrogen, and 200 mL of freshly dried toluene was added by cannula. The flask was subjected to 4 evacuation/backfill cycles, after which the red and gray suspension had turned a deep green/blue, with excess Zn powder and undissolved Cp_2TiCl_2 suspended in the solution. The headspace was evacuated and the flask sealed and left to stir for 2 hours at room temperature, after which all of the Cp_2TiCl_2 had reacted to give a green/blue solution with suspended Zn powder. The schlenk was pumped into the glovebox and the green/blue solution transferred off of the Zn powder and into an erlenmeyer flask by syringe for storage.

To test the glovebox atmosphere:

Syringe 0.3 mL of the Atmoscheck solution into a small vial and place the vial next to the circulation inlet filter. Allow the toluene to evaporate (may take several hours), and record the colour of the crystals/residue left in the vial in the appropriate glovebox log book.

Colour Key:

Green/Blue = GOOD	No further steps required
Yellow/Orange = BAD	Regenerate catalyst column

References:

1) M. Fryzuk, Private Communication
2) Sharon J. Nieter Burgmayer, J. Chem. Ed. **1988**, *75*, 460.

Note: Still tracking down the reference, but apparently Atmoscheck is NOT water sensitive, just oxygen sensitive. Keep this in mind in case of possible water (not air) contamination of glovebox.