Additive Analytics: Easy transformation of low-cost FDM 3D printers for HPTLC sample application
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Step-by-step assembly guide and standard operation information

In Table 1 necessary parts for the modification of a Prusa i3 based 3D Printer are listed. For *italic* written parts CAD files can be downloaded at the following repository webpage: https://www.department.ch.tum.de/wssb/hptlc/

A SLA 3D printer can be used to manufacture these parts in your laboratory or alternatively the nearest CNC machine service or 3D printing service can be instructed.

Table S1: Parts list

| Part   | Description                                                                 | Part   | Description                                                                 |
|--------|----------------------------------------------------------------------------|--------|----------------------------------------------------------------------------|
| 1      | „RepRap“ FDM printer: e.g. Monoprice: i3 Maker Select 3D Printer v2, CA, USA. Prusa Research: Prusa i3, USA | 8      | Solenoid valve, JetCat fuel vent Ingenieurbüro CAT M. Zipperer GmbH, Germany |
| Part 2 | a: Syringe: P/N: 003050, 004050, 005050 (Trajan Scientific and Medical, Melbourne, Australia) b: Swivel nut, c: Extended Plunger | 9 a,b  | a:*Syringe_quickrelease*, b: *Plunger_fix*                                       |
| Part 3 | a,b,c *Syringehandler*.stl a: Aluminium CNC, b: Resin (SLA Printed)          |        |                                                                                |
| Part 4 | Stepper engine GT2 geared NEMA 14                                           | 11     | Nylon hose, 4 mm                                                               |
| Part 5 | Cylinder pins, DIN 7,5 x 32 mm                                              | 12     | Resistors, 50 Ohm, etc.                                                        |
| Part 6 | a: GT2 closed loop belt, 100 mm, b: GT2 pulley                              | 13 a,b | Injection needle (truncated/leur), 1.5 mm                                       |
| Part 7 | a: “atomizer”, b: “print_atomizer”                                          | 14     | “TLC_bed”.stl                                                                   |

Please take note:

Syringes and especially needles can result in harm to your body. Always apply adequate safety precautions. No responsibility can be taken for health risks associated.
Step 1, 3D Printer:

Generally any 3D printer with a sufficient height and bed size can be used as HPLTC tool in conjunction with the sheet needle atomizer. The here presented parts are designed to fit 20 x 10 cm HPTLC analytical plates and an i3 Prusa based open source printer. A version of the recommended low-cost rigid metal frame Monoprice Maker Select 3D Printer v2 is depicted (Figure S1) (Monoprice, Inc. Rancho Cucamonga, CA, USA.).

Step 2, Syringe:

Autosampler glass syringes (Trojan 25, 50, 100 µl, RN) with a removable needle (Figure S2) can be used to apply different sample volumes. Unscrew the cap-nut (Figure S2, Part 2b) and let a milling machine service thread it. Alternatively attach and seal the 3D printable Luer-Lock attachment (Part 7b, not shown).

Step 3, Syringe:

Assemble Spray tool: Atomizer/ Machined screwcap-nut part (Figure S3, Parts 7a or b) and 1.2 mm Needle cannula (Figure S3, attach Part 13a)

Step 4, Syringe assembly:

Assemble sheet-needle sprayer part and SGE syringe (Figure S4). Shorten needles to the same length by using a cutting disc.

*Elongate the SGE plunger with a 1.1 mm cannula by hot soldering. Insert the elongated plunger to the syringe (Figure S4, Part 2c*).
Supporting information

Step 4b, Printable Atomizer:

Optional: use the 3D (SLA/SLS) printable connector Part 7b in conjunction with a standard Leur-Lock syringe-needle with a suitable diameter.

Step 5, Syringe-handler:

Manufacture Part 3 (Figure S5) by SLA (or other) 3D printing or CNC machine methods. Add threads and cut open all holes by a handheld drilling machine when necessary (e.g. Dremel).

Step 6, Syringe-handler assembly:

Assemble GT2 pulley (Figure S6, Part 6b) to Nema 14. When necessary add nudge to drive shaft to increase slip resistance force.

Step 7, Syringe-plunger:

Connect syringe plunger fixture (Figure S7, Part 9b) to the GT2 loop belt (Figure S7). Using a leather punch, create a hole in the loop belt. Add a drive belt spring when necessary.

Alternative solution:
Use a non-looped GT2 belt. Punch two holes at a distance of 200 mm from the centre to centre. Connect both ends with Part 9b (Figure S7).
Step 8, SyringeHandler assembly:
Figure S8, first, put the GT2 looped belt (Figure S8) to the cavity in Part 3a/b.

Step 9, SyringeHandler assembly:
Figure S8, Part 6b should be facing to the bottom side of Part 3a (Figure S9).

Slide in the syringe assembly (Figure S10) into the holder (Figure S9, Part3a).

Step 10a, Syringe fixing:
Clamp syringe into place by adding the syringe quick-release (Figure S11, Part9a).
Step 10b, Syringe fixing:

Use 4 x M2.5 x 33 mm to mount Nema 14, Syringe and the quick-release (Figure S12).

Step 11, Belt:

Use the cylindrical pin as a shaft for the GT2 geared free pulley. (Figure S13, Part 6b). First, add pulley to drive belt. After that add cylindrical pin into the 5.0 mm through-hole.

Hold pulley in place with a 2 mm Allen key (Figure S14). Force the cylindrical punch (Figure S13, Part 5) (DIN 7) trough the pulley.

The slight tension will keep the shaft in place (Figure S15). Should shaft loosening be a problem, add a piece of Duct-tape to the sides of the shaft. Add a small amount of high grade drilling oil as a lubricant to the pulley.
Step 12, Attachment:
Attach TLC Printing head to the linear bearing of the X-axis.

1] (Figure S16, I) Use appropriate set-screws, to fasten TLC sample handler to the X-axis.

2] (Figure S16, II) Depending on the linear bearing type, minor modifications might be required.

3] (Figure S16, III) M4 threaded holes can be used to assemble and disassemble system for syringe exchanges. Verify dimensions before drilling!

4] (Figure S16, IV) Plunger needs to be connected to the drive belt. Therefore move the drive belt to the lowest possible position and push the plunger to its 0 µl position after that fasten with a M1 setscrew. Then note the maximum volume, which can be withdrawn by the drive belt arrangement.

5] (Figure S16, V) Attach the X-axis end stop flush to the Stepper motor for axis zero point setting. Verify the position of the needle tip reaching the outer boundary of the 20 cm HPTLC plates.

6] (Figure S17, VI) Connect NEMA 14 to the previous extruder stepper. Test if a free movement of the extruder and syringe is possible by using the printers manual movement mode. No firmware changes are required.

7] (Figure S17, VII) Reposition the end stop switch of the Z-axis not to be lower than the lowest HPTLC plates surface.
Step 13, Belt:
Connect the geared belt of the X-axis to the syringe handler, if necessary shorten belt. Connect towline cable drag chain to the side of the syringe holder (Figure S18, Part 10b). Check free movement.

Step 14, Nitrogen flow:
Connect solenoid (Figure S19, Part 8) hose to sprayer (Part 7) and Nitrogen gas source. Connect the power cable to previous 3D printers cooling fan. Add a resistor (e.g. 50 Ohm) to restrict the load. (Figure S19, Part 12)

Step 15, Bed:
Add HPTLC plate holder (Figure S20, Part 14) to the previously heated printing bed. Remove temperature sensor and exchange with a constant resistor if firmware requires head bed temperature parameter (10k Ohm). Remove heat pad power cable and upcycle.
Standard operating tips:

1. Never start a sampling process when the machine is unobserved and accessible to uninstructed colleagues.

2. Never shift positions of plates or samples or solvents when the printer is in operation. Pause or switch off, and continue after corrections.

3. The glass syringe is intentional the weakest part of the printer, should the GCODE do not match the physical setup this might lead to breakage of the glass cylinder. Which typically minimises further mechanical damage.

4. When using toxic organic solvents, place the device in a suitable hood.

5. Connect hose at a pressure of 2 bar nitrogen gas to the solenoid valve connected to the sprayer.

6. In order to apply samples, the 3D printer movements need to be set up to run a sequence of liquid handling operations. Make sure to program the right movements to the printing head. A sample GCODE sequence is added at the bottom of this document.

7. Before starting to apply samples, the syringe needs to be cleaned first, and in between samples. Therefore the machine needs to move the syringe to a vial filled with a suitable organic solvent. Engage the flow of nitrogen during dispensing.

8. Depending on the solvent used, spray times must be adjusted to ensure drying of the samples on the plate.

9. Seal samples with a pierceable tape, reducing solvent evaporation during operation. 96 Well plates are suitable as a sample container.

10. Add up to 8 Glass plates to the 20 x 20 cm printing bed. When using 16 lanes per plate, 4 controls, and 12 Samples, 96 samples can be processed at once. If samples are susceptible to rapid oxidation, a stream of nitrogen can be connected to the back of the rack when using a two-way solenoid.

Sample application checklist:

- Open the nitrogen flow to the solenoid
- Manually push the plunger to 0 µl
- Visually level bed and Z-axis by movement of the nut on endless screws
- Add (washed) HPTLC plates
- Add (open or sealed) liquid samples
- Add wash solvent volume required (~100µl per sample)
- Add Kim wipe to waste vail to absorb solvent fog
- RUN correct GCODE

Please cite as: (Woortman et al. 2020c)
Numerical script guide GCODE

GCODE commands can be written in any text editor. When saving the text file with the .gcode file extension, these will be recognized by any FDM 3D Printer. For further information see: https://reprap.org/wiki/G-code

An commented example can be found below:

Step 1,
Coordinate system reset

Initially, the cartesian coordinate system is zeroed with the help of end stop limits.

Take note:
The syringe plunger must be in its lowest position when starting the script/print.

Step 2,

1. Sample application, by initially moving the needle tips to the sample vial, withdrawing sample with the extruder (E).
2. Drying the dual needle before sample application by a burst of nitrogen is recommended!

Nitrogen/Solenoid
ON (M106 P2 S255).
OFF (M106 P2 S0).

4. Sample application, with solenoid/nitrogen flow ON (moving back and forth for improved drying and atomization)

These parameters can be altered to fit specific sample volume and solvent needs.

Step 3,

Needle wash steps, required to clean syringe syringe and needle tip.

Use adequate waste vials