

A self-made tube cracker, coupled to an EA-IRMS-AGE3 system

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Online supplementary information

Cracker operation protocol

System preparation

1. In the EA software: Options => Maintenance => Replace Parts
2. Disconnect the quartz bridge and connect the cracker device.
3. Arrange cracker valves to 'EA Only' mode (1C, 2C, 3B, 4B).
4. Connect an external He cylinder to valve 3. Set reducer pressure to 1.5 bar. If there is a valve on that line, open it partially.
5. In the EA software, click 'Finished' and confirm.
6. Run RunIn samples till C and N areas are below 100.
7. Change valves 3,4 to position C ('Washing mode').
8. Wait 1 minute. Verify He washes on valve 4.
9. Change all valves to 'Collection mode' (all valves to position A. Change valve 4 first, then 3, and then 1 and 2 simultaneously).
10. Run RunIn samples till C and N areas are below 100.
11. Run Daily tuning in IRMS.
12. Verify that Auto (in AGE) is not on. Start Conditioning.
13. Insert sample list to the IRMS ionOS software with a RunIn between samples. For the samples, the EA Method specified below works well for us. It includes 10s of oxygen only as no sample is burnt. RunIns should be with 120s as usual. Include 2 IRMS standards before and another 2 after the samples. Include EA-IRMS Sleep lines at the end.
14. Change valves to 'EA Only' mode (1C+2C simultaneously, 3B, 4B).
15. Run the first 2 IRMS standards manually in EA-IRMS mode.
16. Scratch the glass tubes for easier cracking.

Measurement

1. Schedule all samples in IonOS. Have a RunIn as the first line. If a 'Pre' sample exists, have a RunIn after it.
2. Once the Conditioning is finished, press 'Auto', 'Remove H₂O', and '+IRMS' on.

3. Click 'Sample' in AGE, then 'Yes', and 'Continue'. The system should start running the first RunIn.
4. While RunIn is running (on 'EA Only' mode), open the cracker and insert a tube (after the tube was scratched for cracking). Close the cracker.
5. Fix the brace into position.
6. Change valves 3 and 4 to position C ('Washing mode'). Make sure He is flowing out through valve 4.
7. Wait 2 minutes.
8. Stop washing by changing valves 4 and 3 to position B (first 4 and then 3).
9. At the end of the RunIn, AGE will clean the trap.
10. Wait for Trap Cleaning process to cool down and reach 120°C.
11. Change valves to 'Washing mode' (3 and 4 to positions C) and wash the line with He.
12. When the temperature is 80°C, stop washing by changing valves 4 and 3 to position B (first 4 and then 3).
13. Bend the cracker and break the tube.
14. Change all valves to 'Collection mode' (all valves to position A. Change valve 4 first, then 3, and then 1 and 2 simultaneously). This should be done before the EA-IRMS starts to take the sample itself.
- 15. The sample should be collected and loaded into its reactor.**
- 16. When the next RunIn starts, switch the valves to 'EA Only' mode (first 1C+2C simultaneously, then 3B, 4B).**
- 17. Replace the tube in the cracker with a new one. Make sure to scratch the glass tube before insertion.**
- 18. Fix the brace into position.**
- 19. Change valves 3 and 4 to position C ('Washing mode'). Make sure He is flowing out through valve 4.**
- 20. Wait 2 minutes.**
- 21. Wait for Trap Cleaning process to cool down and reach 120°C.**
- 22. Change valves to 'Washing mode' (3 and 4 to positions C) and wash the line with He for several seconds.**
- 23. When the trap temperature reaches 80°C, stop washing by changing valves 4 and 3 to position B (first 4 and then 3).**
- 24. Bend the cracker and break the tube.**
- 25. Change all valves to 'Collection mode' (all valves to position A. Change valve 4 first, then 3, and then 1 and 2 simultaneously).**
26. Repeat steps 15-24 until all samples are loaded to the reactors. Verify that the reaction is starting.
27. When the RunIn after the last sample starts, change valves to 'EA Only' mode (first 1C+2C simultaneously, then 3B, 4B).
28. Insert 2 IRMS standards into the EA carousel.

EA Method parameters:

O2 dosing time: 10s

Autozero delay N: 10s

Autozero delay C: 16s

Peak anticipation N: 60s

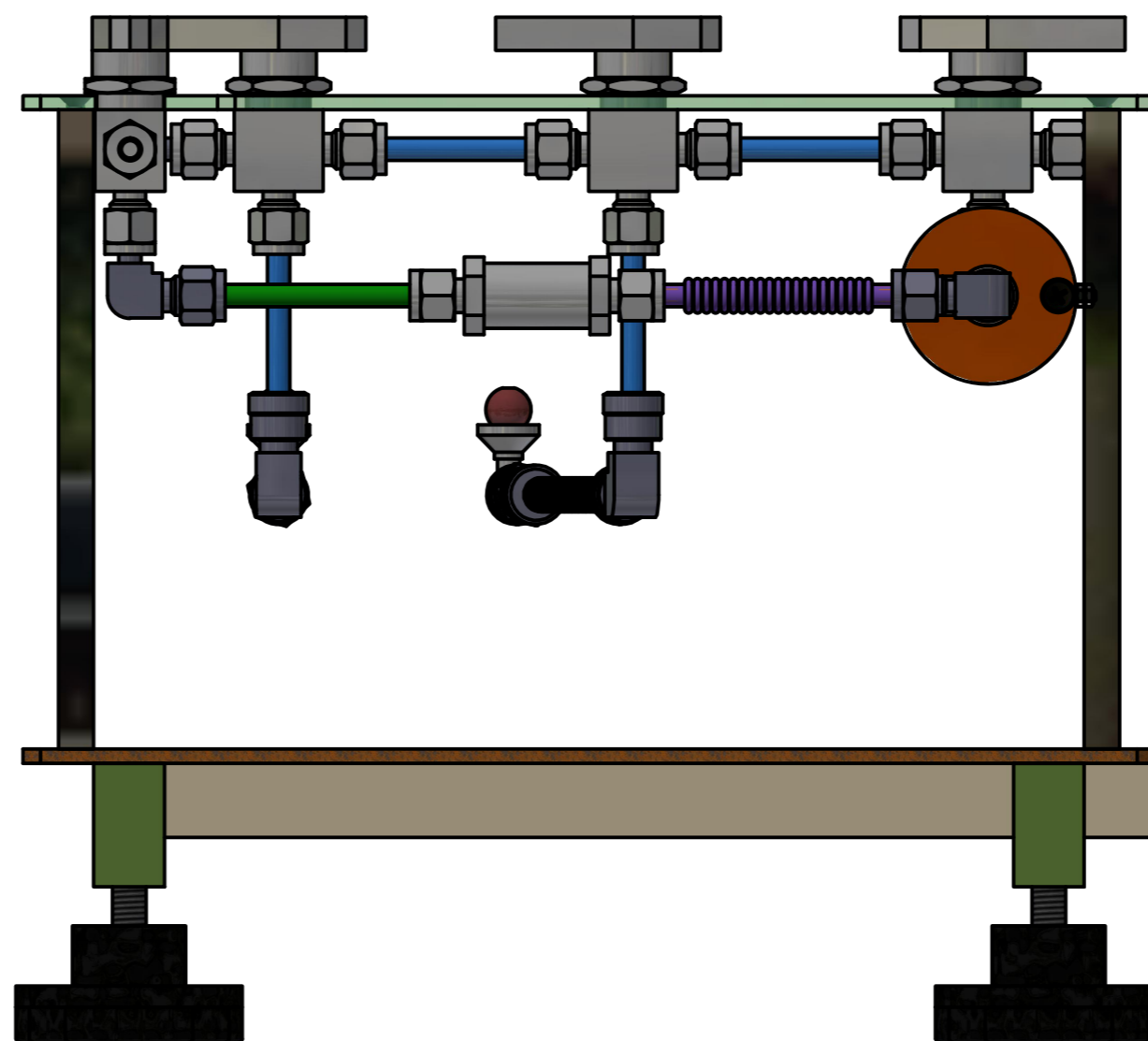
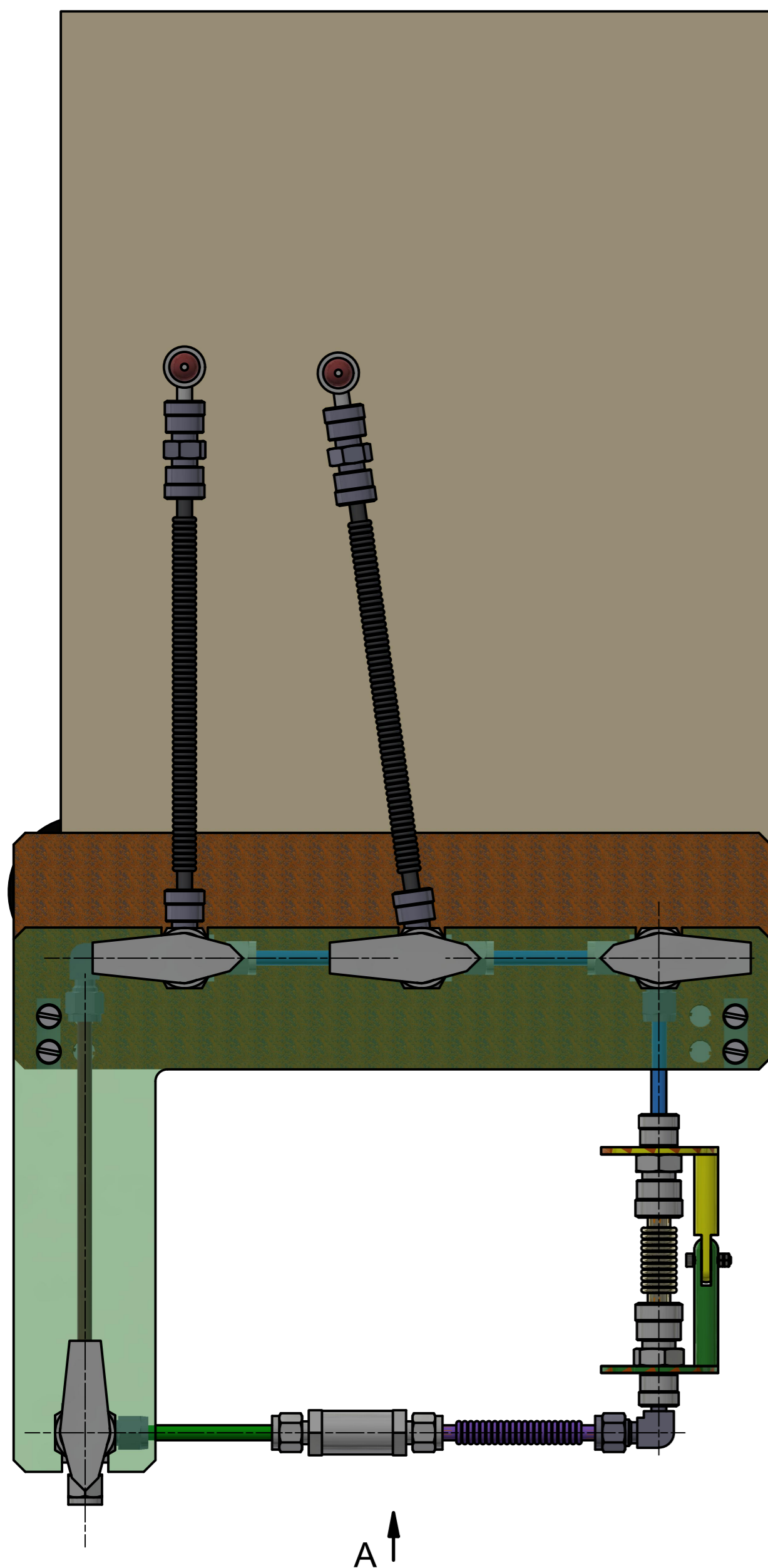
Peak anticipation C: 170s

Desorpt. CO2: 70°C

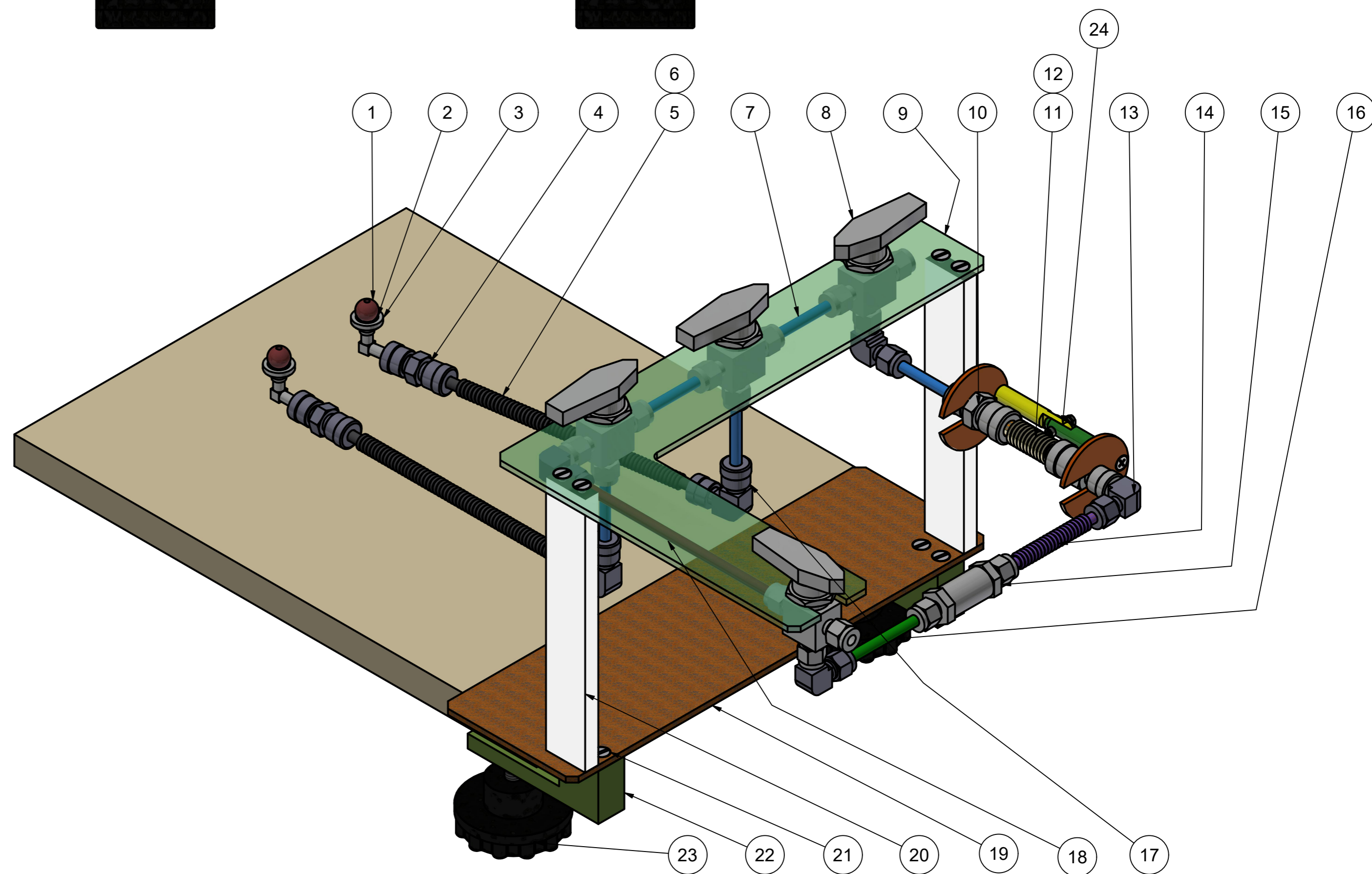
IRMS Method parameters:

Only the peak durations were changed. The N peak duration was shortened by 45 seconds, and the C peak duration was extended by 45 seconds.

Note that the above settings will probably require some adjustment, as not all system components are identical everywhere (such as He flow, length of pipes, etc.).



A (1:2)

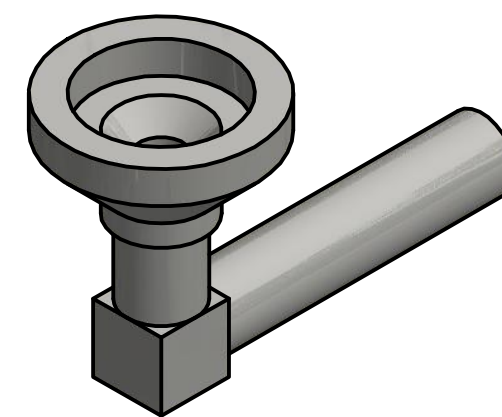
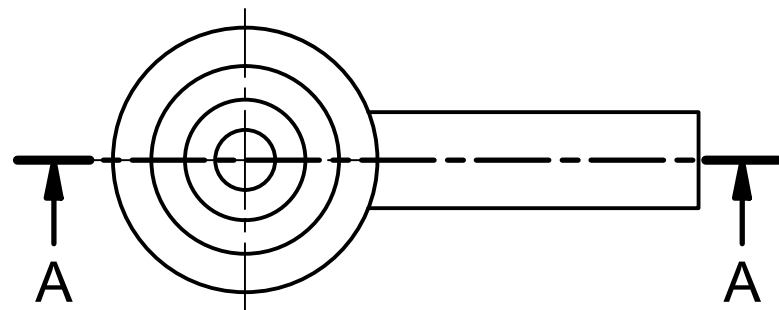
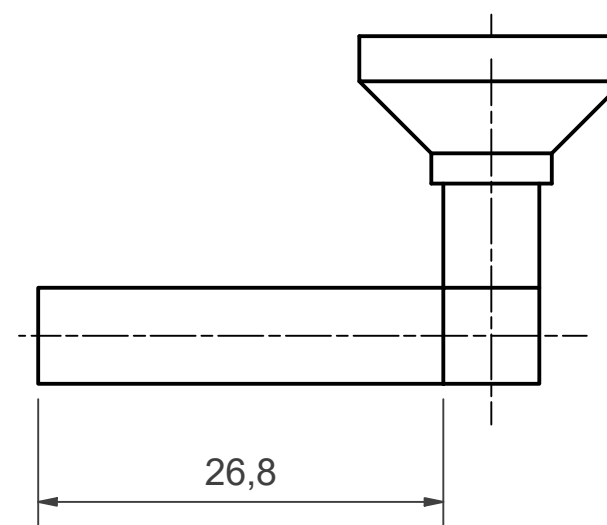
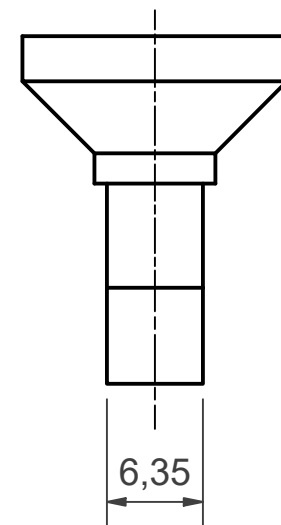
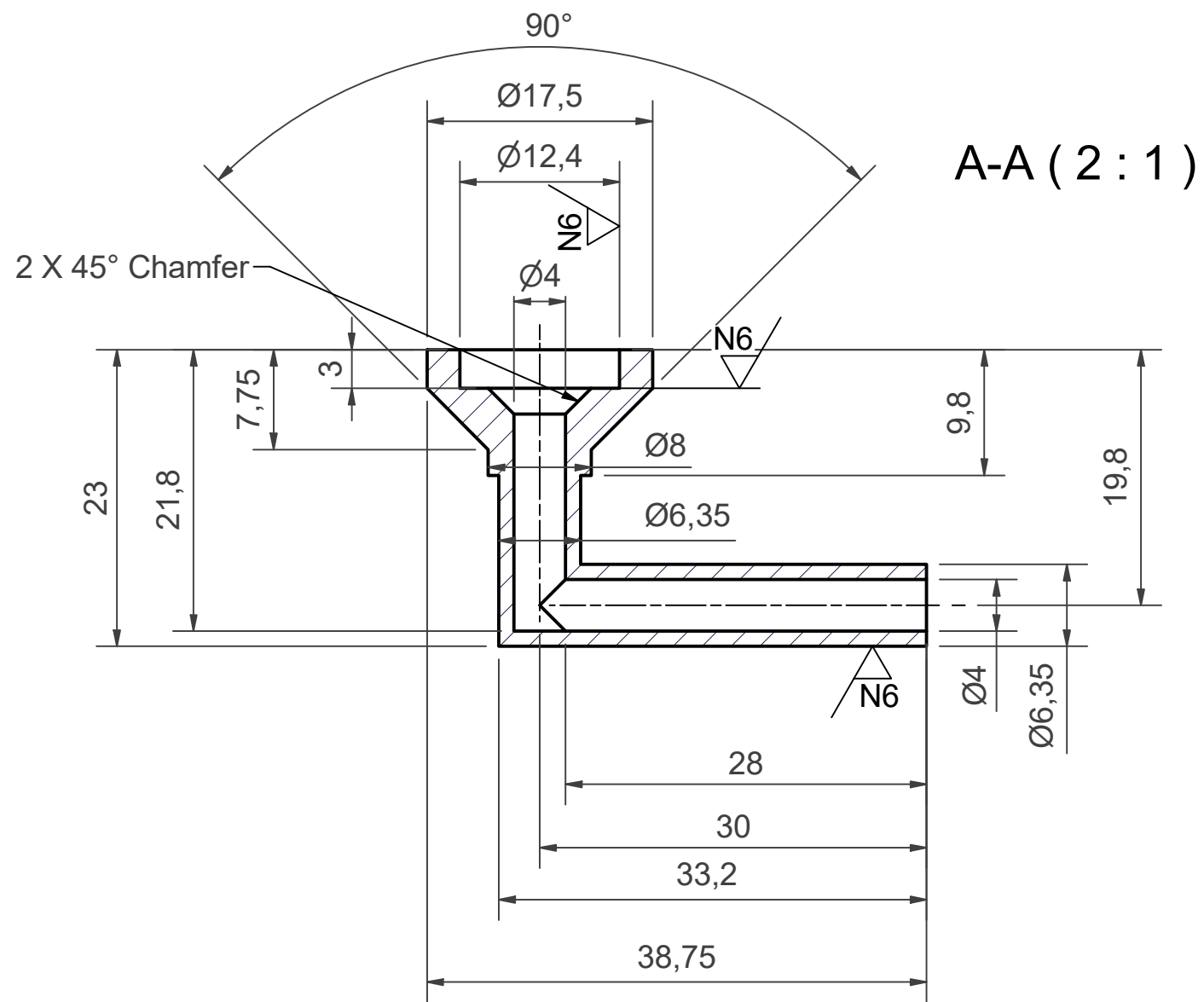


Cracker for D-REAMS lab		תכנון מכשירים Instrument Design	Updated by	Date
Ordered By היחידה לארכאולוגיה מדעית		Weizmann Institute of Science	Designed by Lilia	Date 31-Aug-20
Project	Part	Part Name	Material	Quantity
5292.00- Assembly1				

24	Brace	1			
23	Handle	2			ק"מ
22	Clamp	2	Aluminum 6061		
21	Slotted countersunk flat head screw - M5 x 12	12	Stainless Steel		
20	Post	2	Aluminum-6061		
19	Plate2	1	Aluminum-6061		
18	Tube2	1	Stainless Steel 316	Ø1/4" x 0.9 x 173.8	
17	Union Elbow SS-4-UT-9	2	Stainless Steel 316	Ultra-Torr Vacuum Fitting, Union Elbow, 1/4 in. Tube OD	Swagelok
16	Tube3	1	Stainless Steel 316	Ø1/4" x 0.9 x 82.6	
15	Filter SS-4F-60	1	Stainless Steel 316	In-Line Particulate Filter, 1/4 in. Swagelok Tube Fitting, 60 Micron Pore Size	Swagelok
14	Bellow 321-4-X-2	1	Stainless Steel	Flexible Tubing, 1/4 in. OD, 2 in. Nominal Produced Flexible Length	Swagelok
13	Reducing 90° Elbow SS-400-2R-4	4	Stainless Steel 316	Tube Fitting, Reducing 90° Elbow, 1/4 in. Tube OD	Swagelok
12	Adapter SS-304-6-XOA	2	Stainless Steel 304	XOA Adapter for Convolute Vacuum Tubing, 1/4 in. Tube OD	Swagelok
11	Bellow 321-6-X-1	1	Stainless Steel 321	Flexible Tubing, 3/8in. OD, 1 in. Nominal Produced Flexible Length	Swagelok
10	Reducing Union SS-6-UT-6-4	2	Stainless Steel 316	Ultra-Torr Vacuum Fitting, Reducing Union, 3/8 x 1/4 in. Tube OD	Swagelok
9	Plate	1	Aluminum-6061		
8	3-Way Ball Valve SS-43GXS4	4	Stainless Steel 316	1-Piece 40G Series 3-Way Ball Valve, 0.90 Cv, 1/4 in. Swagelok Tube Fitting	Swagelok
7	Tube1	5	Stainless Steel 316	Ø1/4" x 0.9 x 70	
6	Adapter SS-304-4-XOA	6	Stainless Steel 304	XOA Adapter for Convolute Vacuum Tubing, 1/4 in. Tube OD	Swagelok
5	Bellow 321-4-X-6	2	Stainless Steel 321	Flexible Tubing, 1/4 in. OD, 6 in. Nominal Produced Flexible Length	Swagelok
4	Union SS-4-UT-6	2	Stainless Steel 316	Ultra-Torr Vacuum Fitting, Union, 1/4 in. Tube OD	Swagelok
3	Cap	2	Stainless Steel 316		
2	O-Ring	2	Viton		Existing
1	Column Ball	2	Glass		Existing
ITEM	PART NUMBER	QTY	MATERIAL	DESCRIPTION	COMMENTS

Parts List

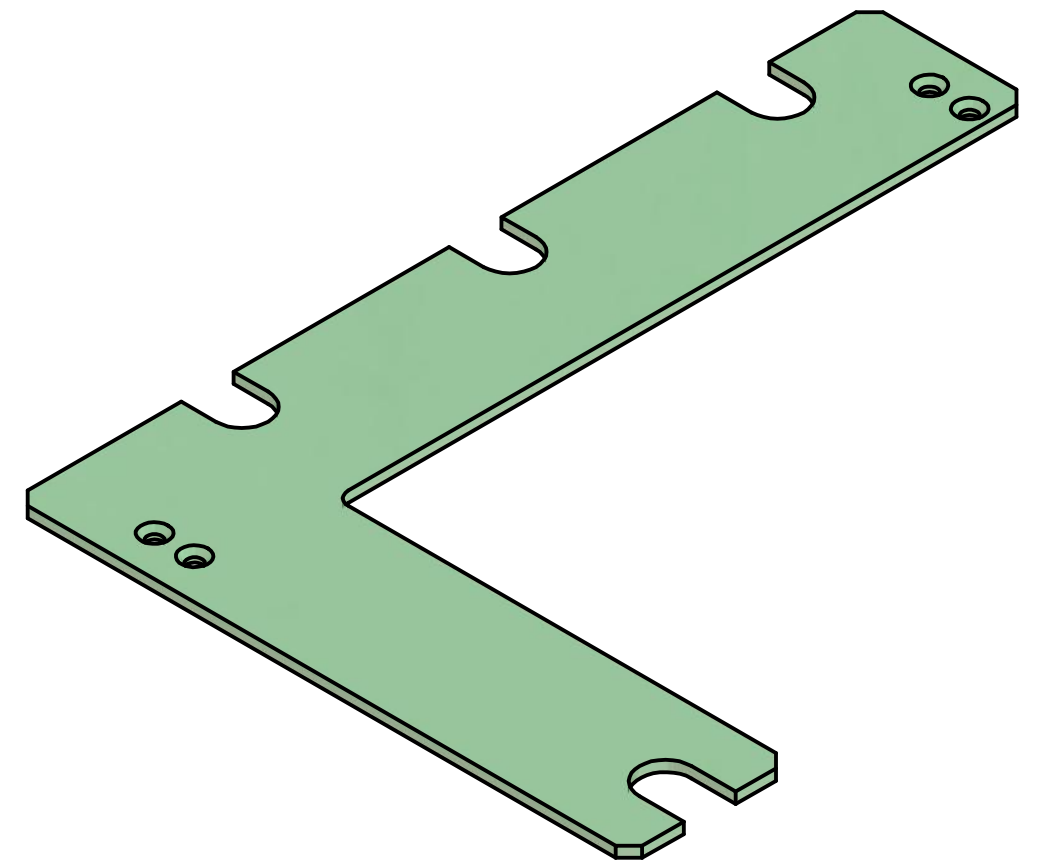
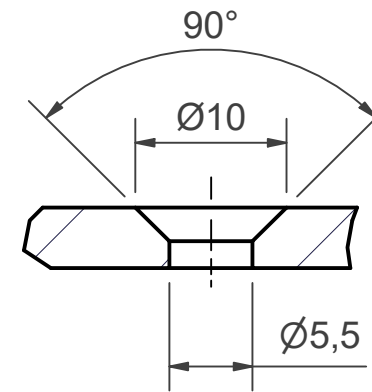
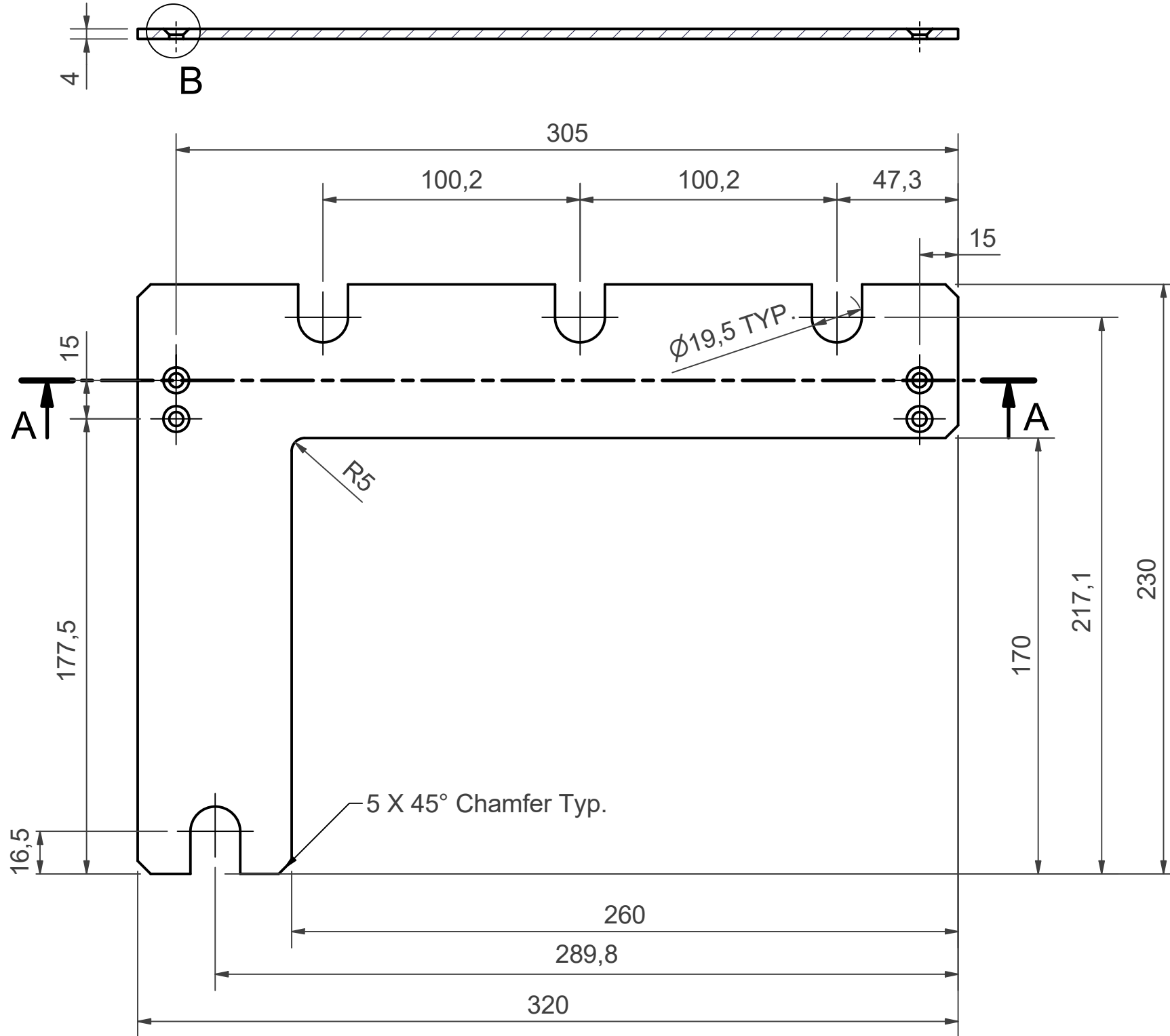
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Project Part Part Name 5292.00- Assembly1		Material		Lilia	31-Aug-20
				Quantity	



Cracker for D-REAMS lab		תכנון מכשירים Instrument Design		Updated by	Date
Ordered By היחידה לארכאולוגיה מדעית		Weizmann Institute of Science		Designed by Lilia	Date 07-Sep-20
Project Part Part Name 5292.00- 03 Cap		Material Stainless Steel 316		Quantity	

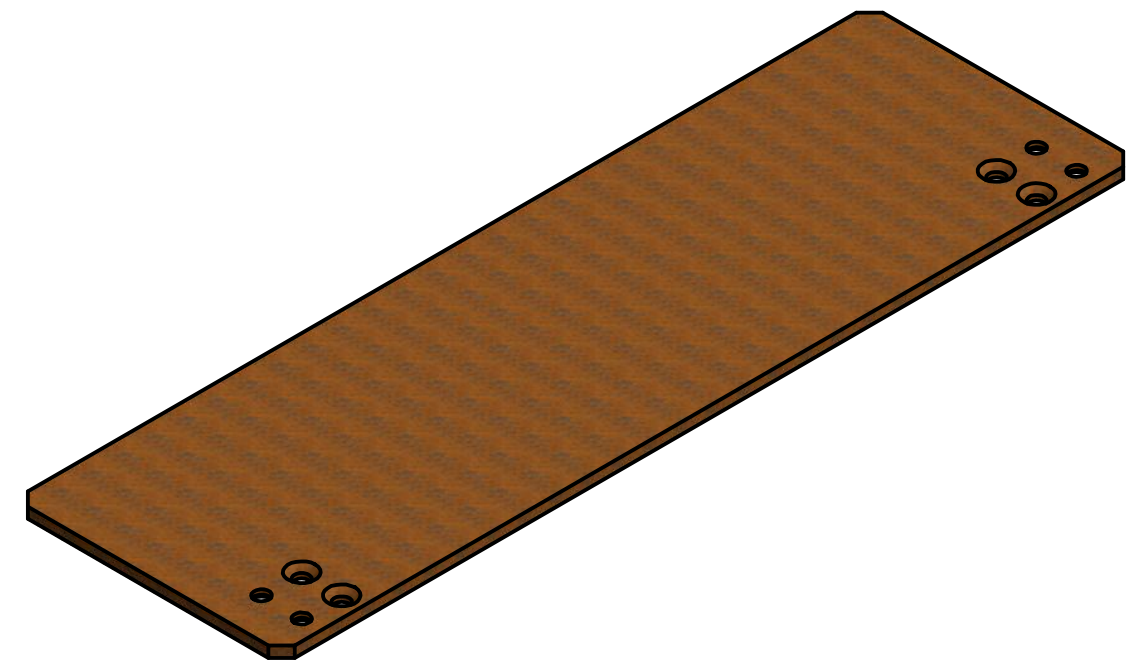
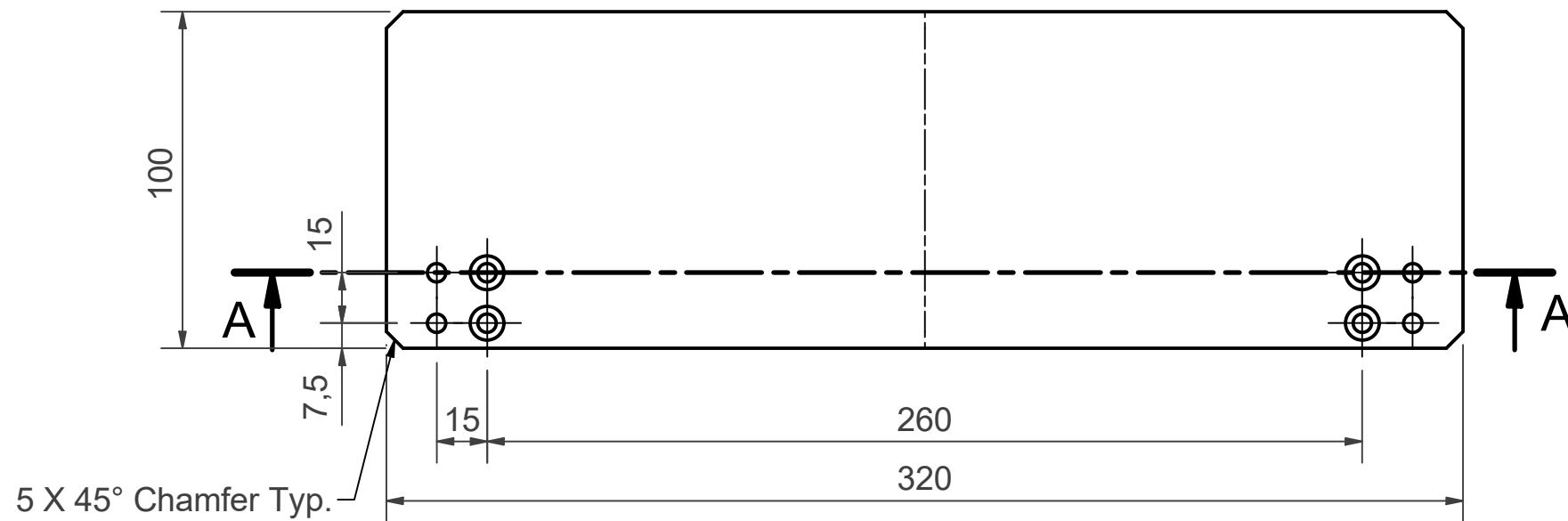
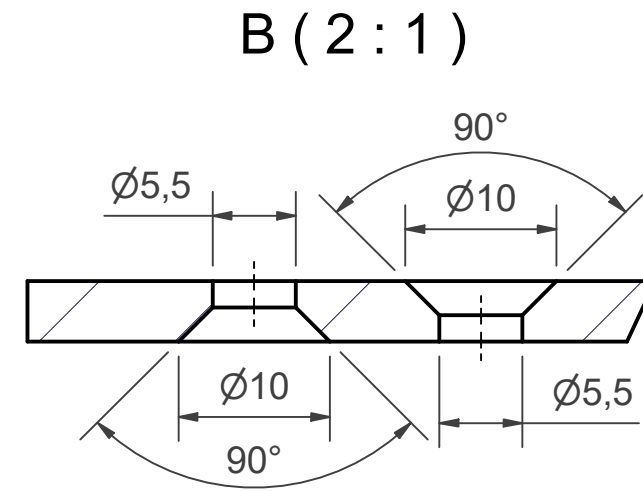
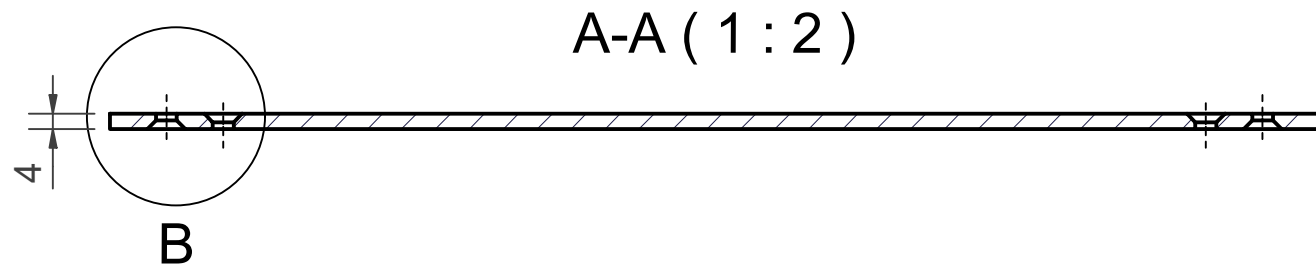
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B (2:1)



Cracker for D-REAMS lab		תכנון מכשירים Instrument Design		Updated by	Date
Ordered By היחידה לארכאולוגיה מדעית		Weizmann Institute of Science		Designed by Lilia	Date 02-Nov-20
Project Part Part Name 5292.00- 09 Plate		Material Aluminum-6061		Quantity	

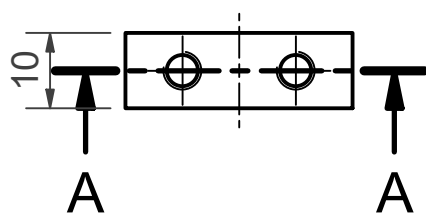
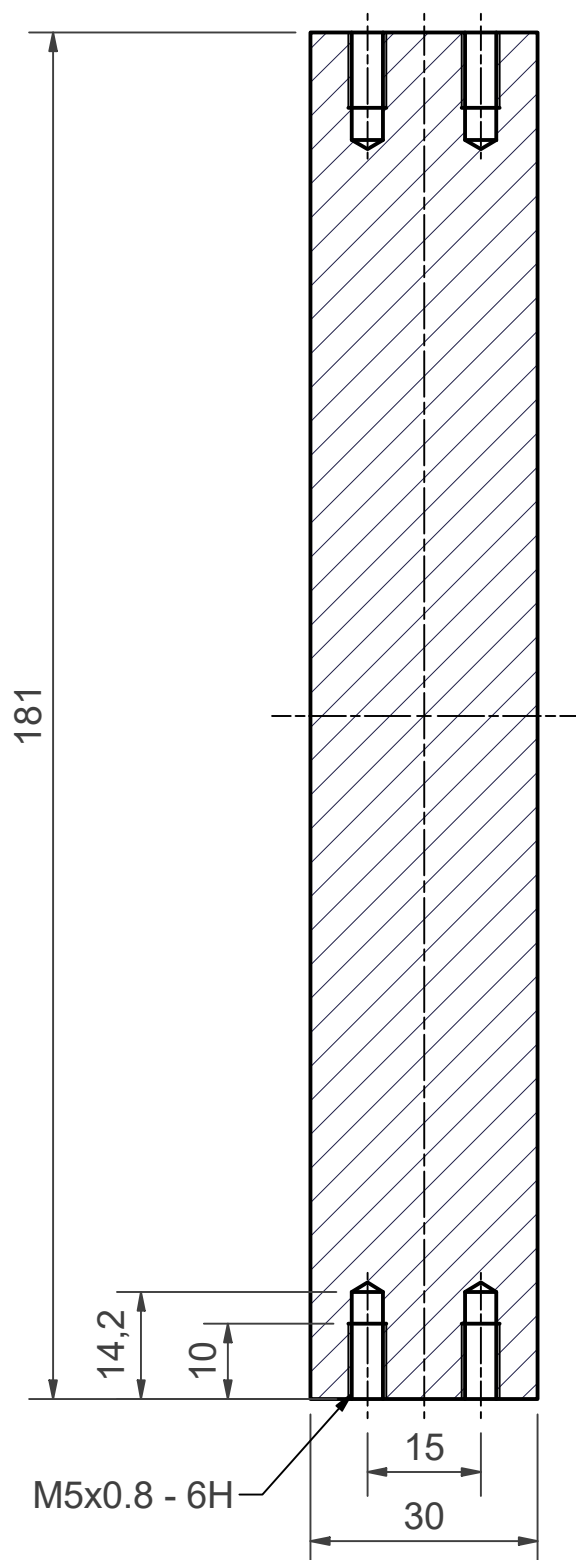
שבור פינות



שבור פינות

Cracker for D-REAMS lab		תכנון מכשירים Instrument Design		Updated by	Date
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Project Part Part Name 5292.00- 19 Plate2			Material Aluminum-6061		Quantity

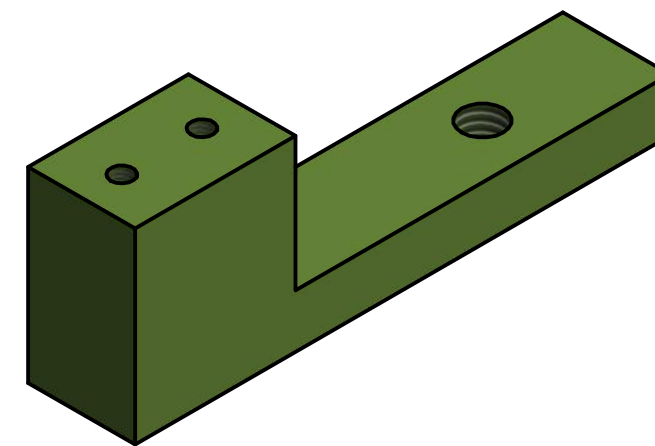
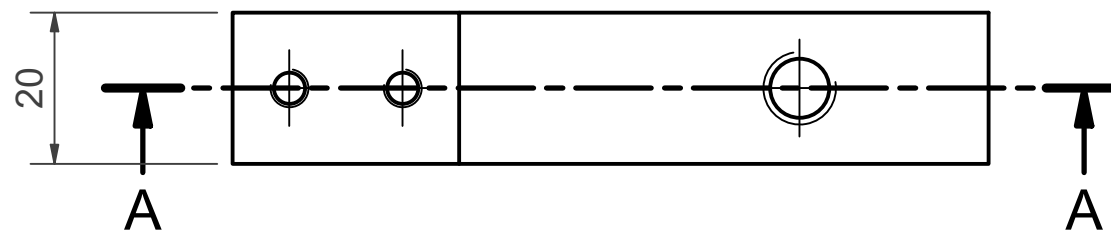
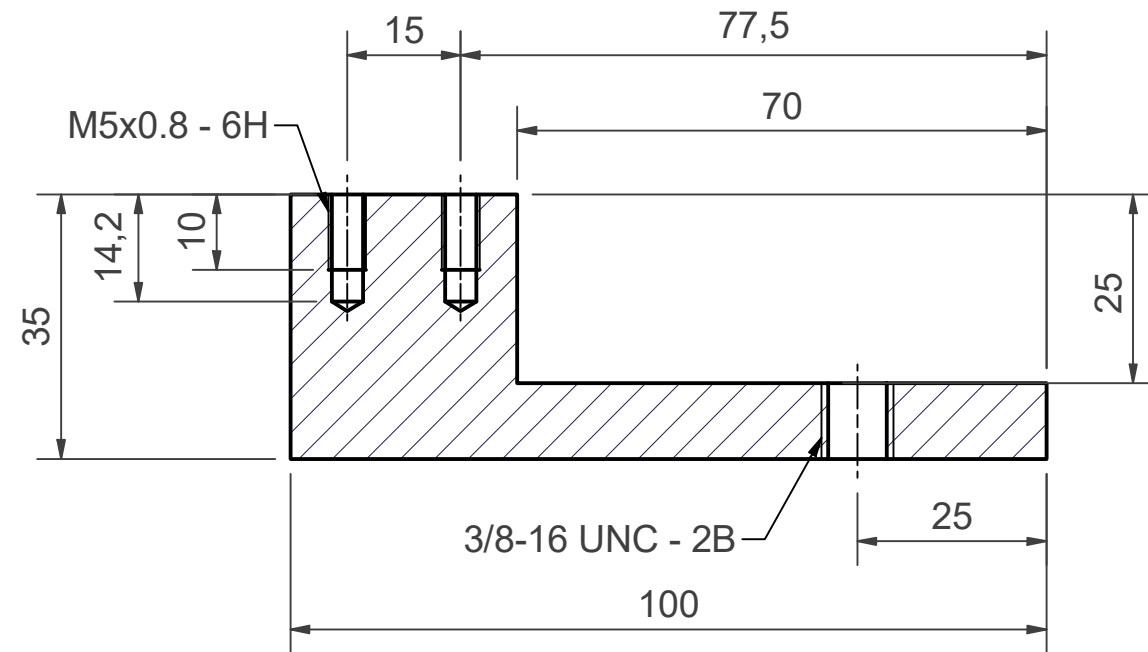
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שבור פינות

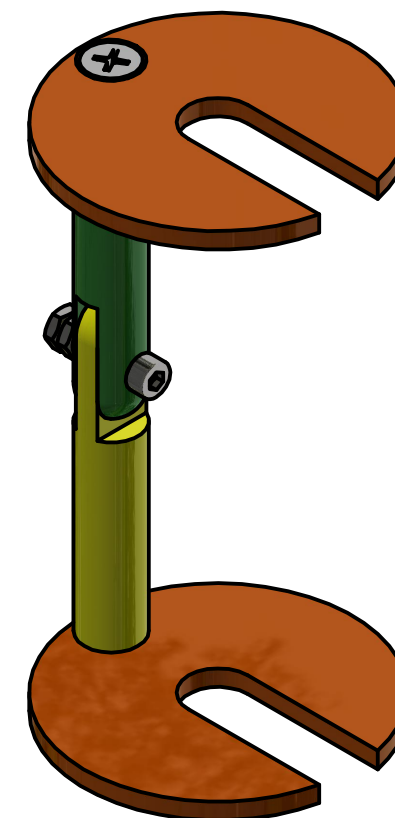
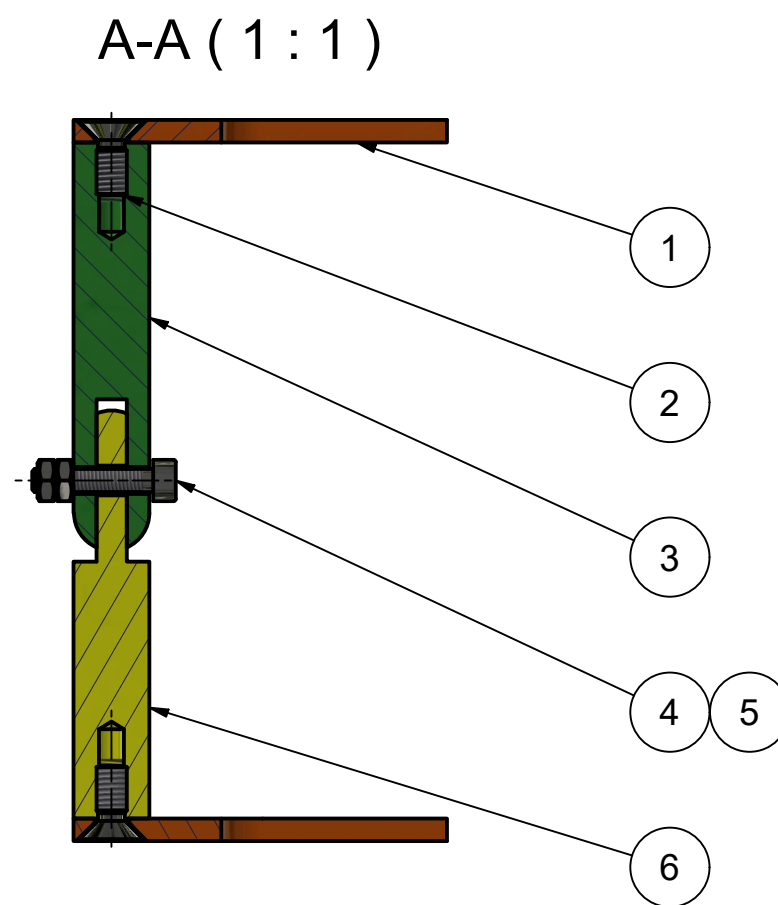
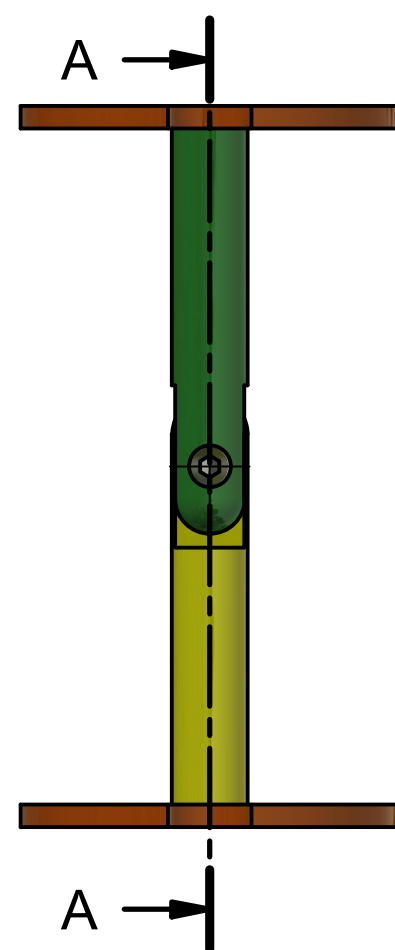
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		Designed by	Date
Ordered By היחידה לארכאולוגיה מדעית	Weizmann Institute of Science	Lilia	02-Nov-20
Project 5292.00- 20 Post	Part Part Name	Material Aluminum-6061	Quantity

A-A (1 : 1)



שבור פינות

Cracker for D-REAMS lab		תכנון מכשירים Instrument Design  		Updated by	Date
Ordered By היחידה לארכאולוגיה מדעית		Weizmann Institute of Science		Designed by	Date
Project Part Part Name 5292.00- 22 Clamp		Material Aluminum 6061		Lilia	02-Nov-20
				Quantity	

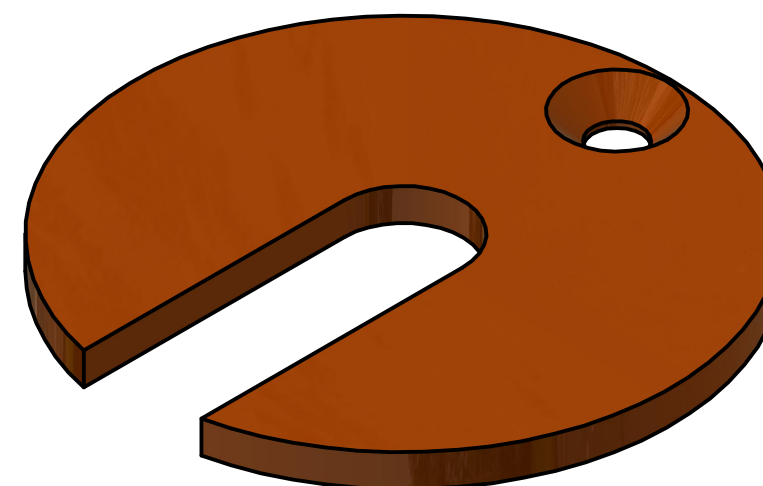
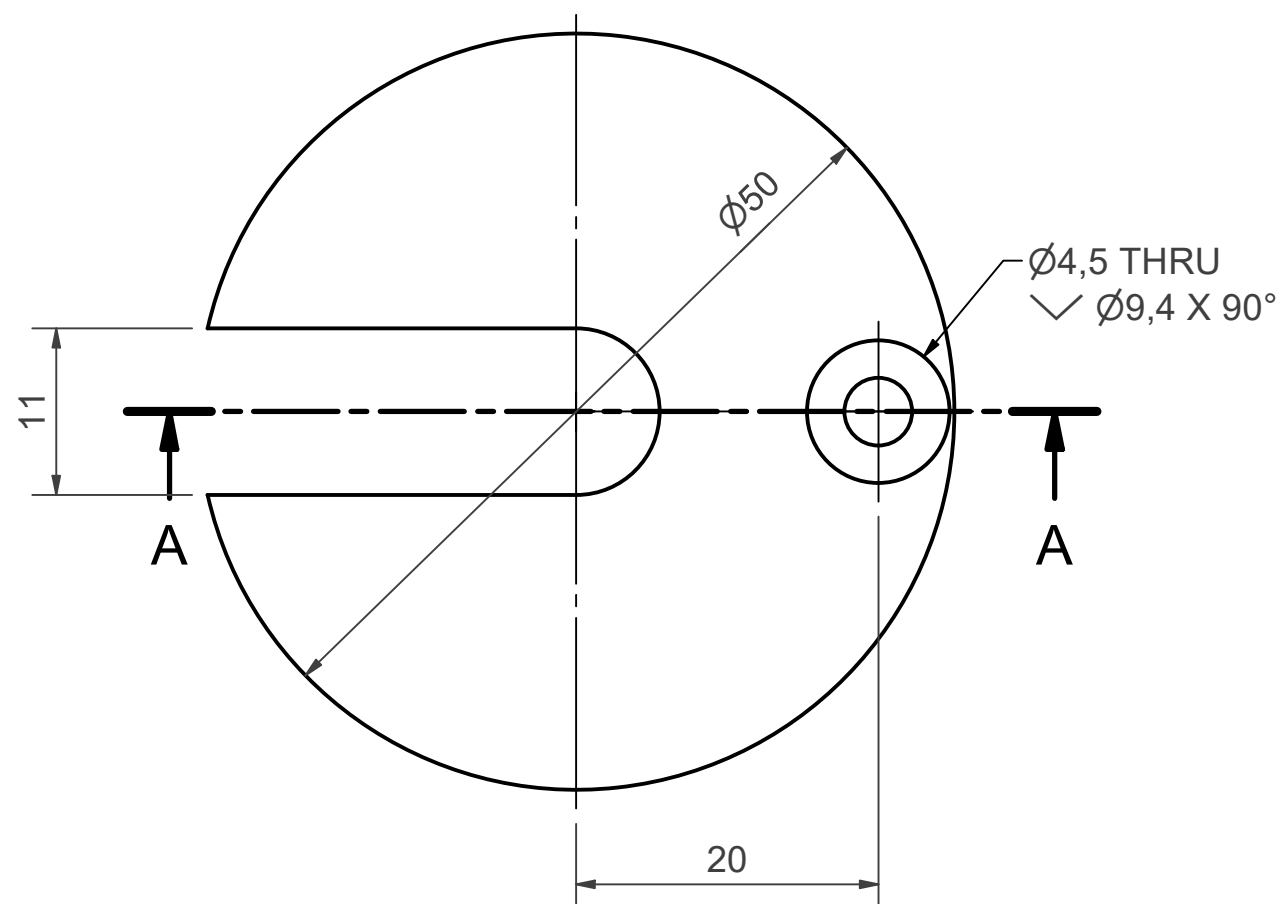
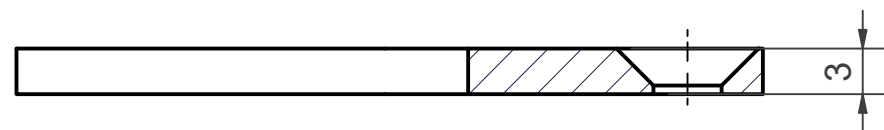


6	Pin2	1	Aluminum-6061		
5	Hexagon nut - M3	2	Stainless Steel		
4	Hexagon Socket Head Cap Screw - M3 x 16	1	Stainless Steel		
3	Pin 1	1	Aluminum-6061		
2	Countersunk flat head screw - M4 x 10	2	Stainless Steel		
1	Disk	2	Aluminum 6061		
ITEM	PART NUMBER	QTY	MATERIAL	DESCRIPTION	COMMENTS

Parts List

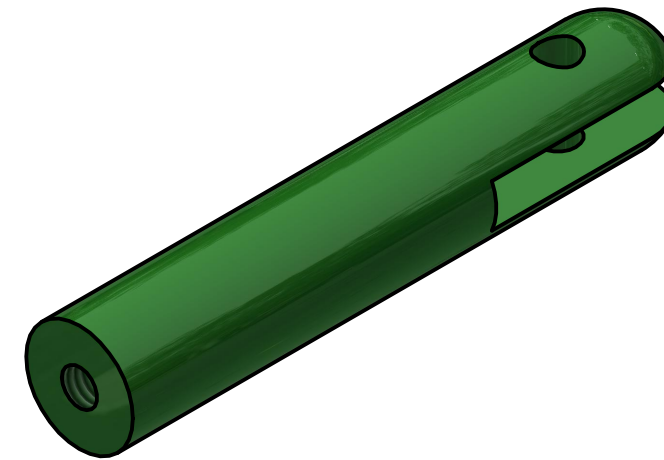
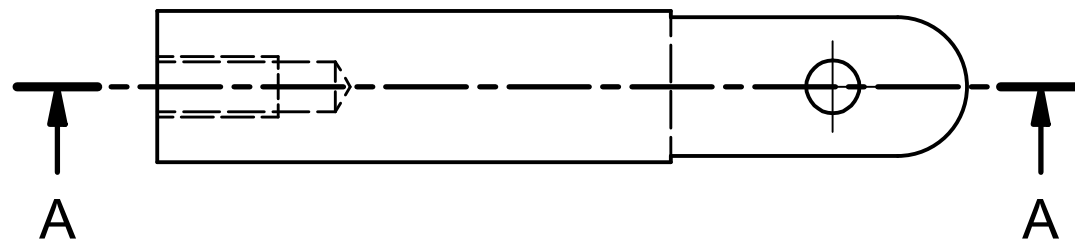
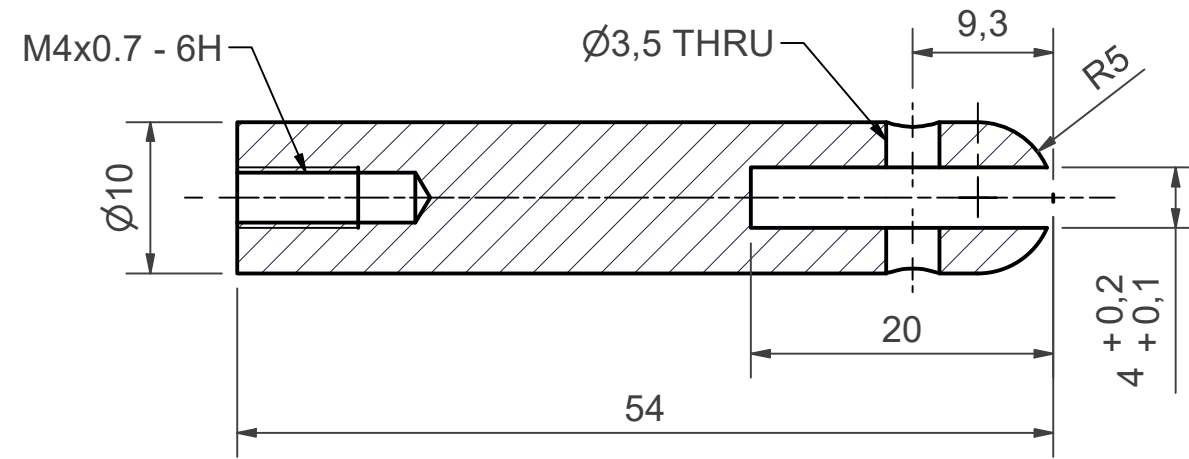
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Ordered By היחידה לארכאולוגיה מדעית		 Weizmann Institute of Science		Designed by	Date
Project		Material		Lilia	03-Nov-22
Part		Quantity			
5292.01-24 Brace					

A-A (2 : 1)



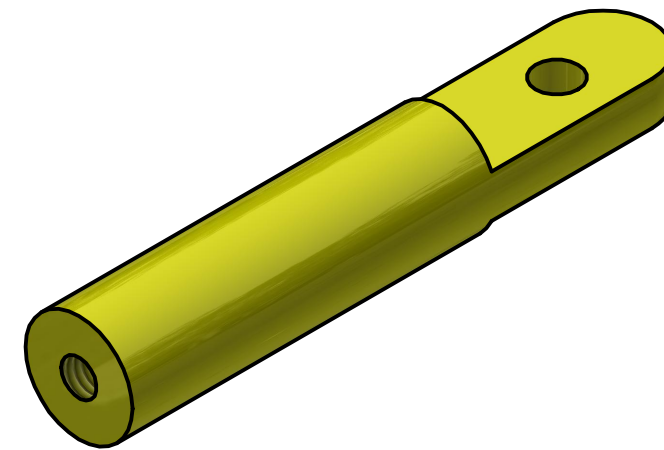
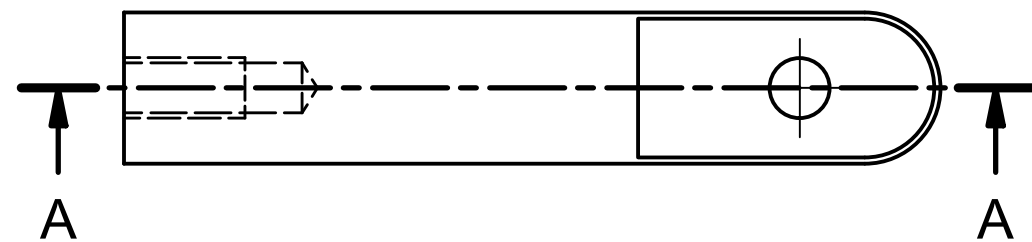
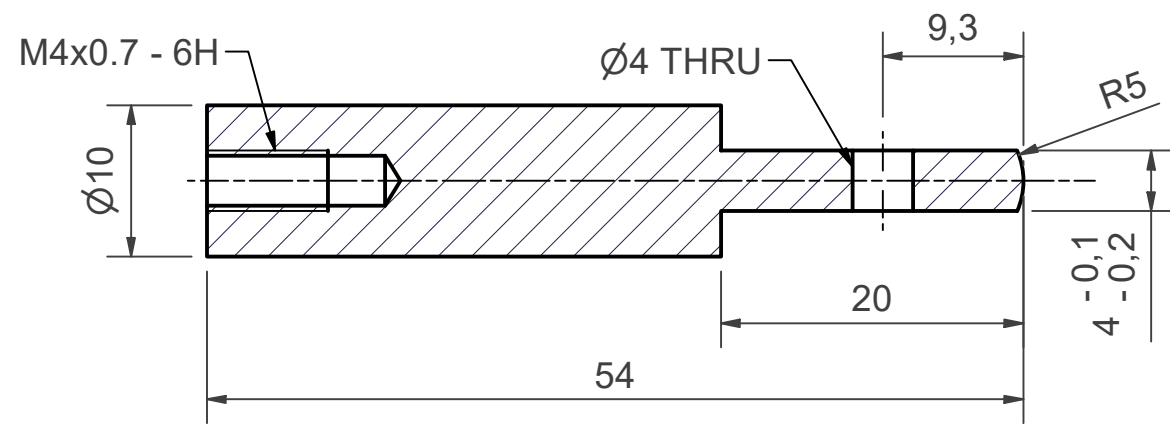
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Project Part Part Name 5292.01- 24-01Disk		Weizmann Institute of Science	Lilia	03-Nov-22
Material			Quantity	
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A-A (2 : 1)



Cracker for D-REAMS lab		תכנון מכשירים Instrument Design 	Updated by	Date
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Material			Quantity	
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A-A (2 : 1)



Cracker for D-REAMS lab		תכנון מכשירים Instrument Design 	Updated by	Date
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