Supporting Information

Boosting energy input and milling efficiency in a mixer mill with 3D-printed milling bodies

Tim Jansen, Daniel John Schmitz, Jovana Obradović and Carsten Bolm*

Institute of Organic Chemistry, RWTH Aachen University, Landoltweg 1, 52074 Aachen (Germany), E-mail: carsten.bolm@oc.rwth-aachen.de

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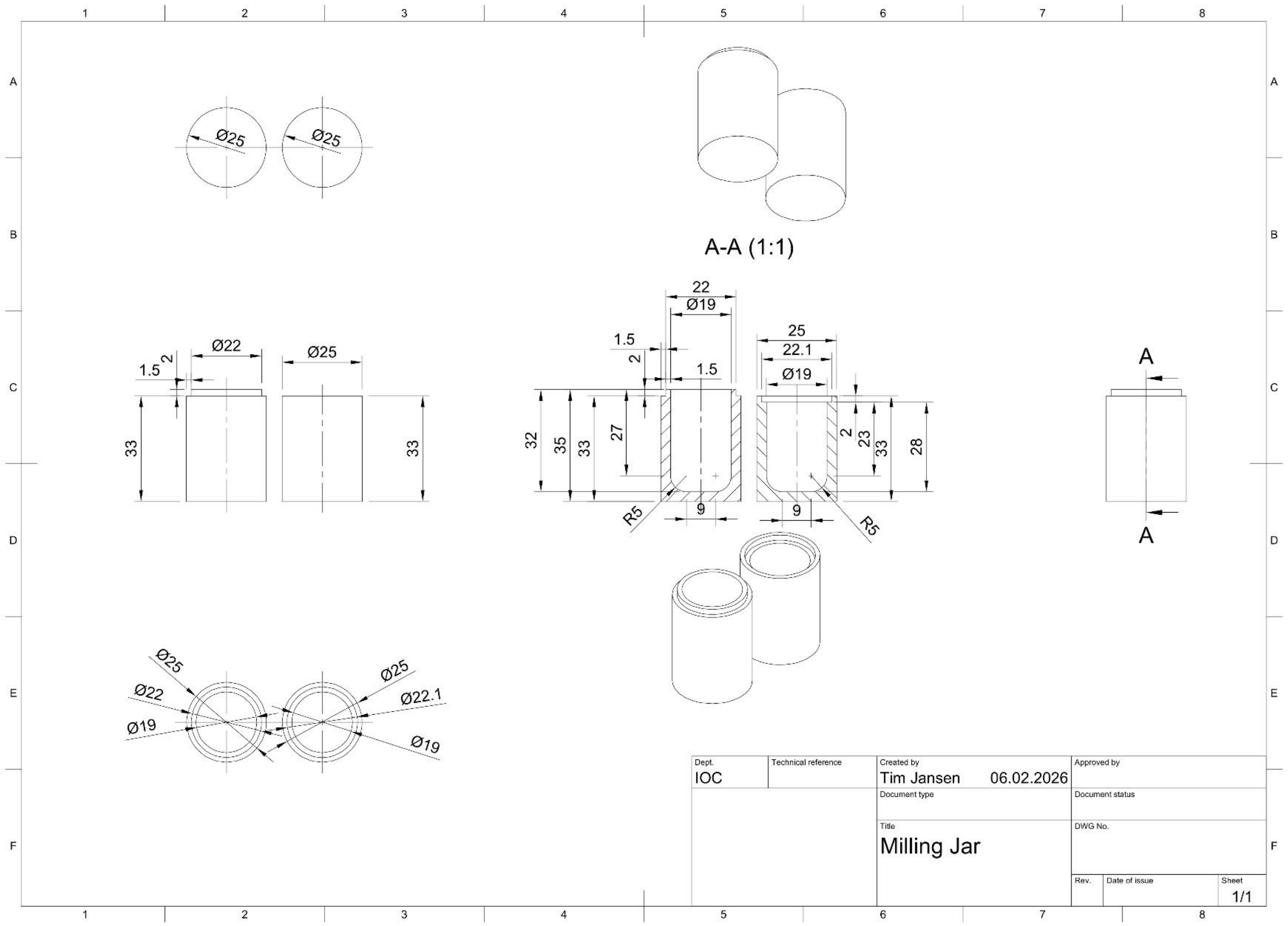
1 Technical drawings of the 3D prints

The 3D-modeling and the technical drawings were done using the software Fusion 360 from Autodesk. The technical drawings of the milling jar and the milling bodies should be printed on DIN A3 and DIN A4 paper, respectively, to get a 1:1 scale. All measurements given are millimeters. For further information about the technical drawings, details and 3D files contact us.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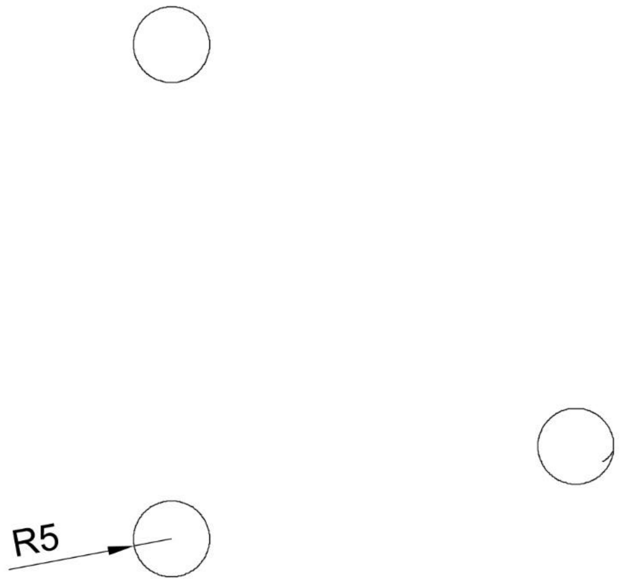
Milling Jar

S4



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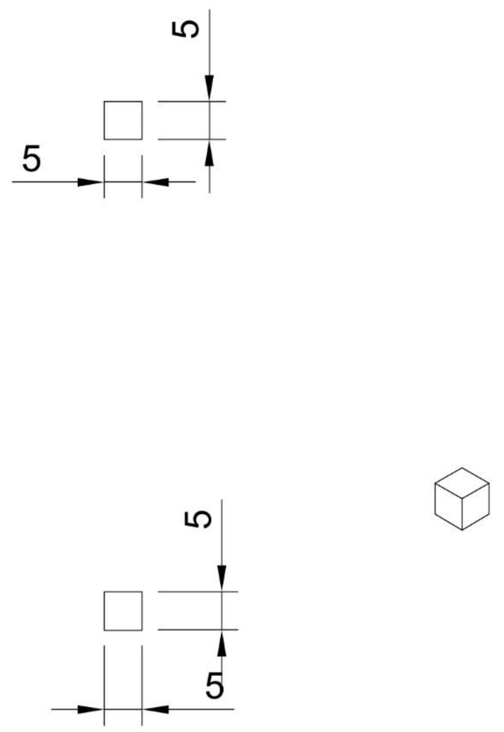
Milling Ball



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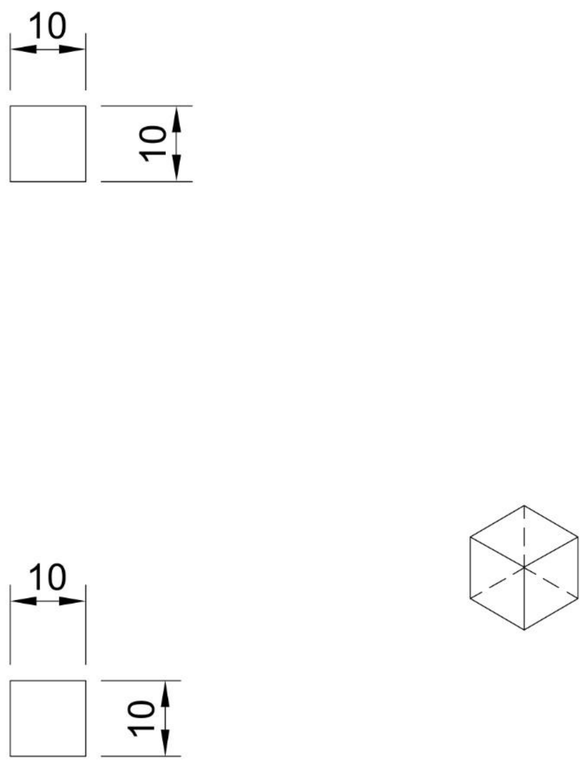
Cube 5mm



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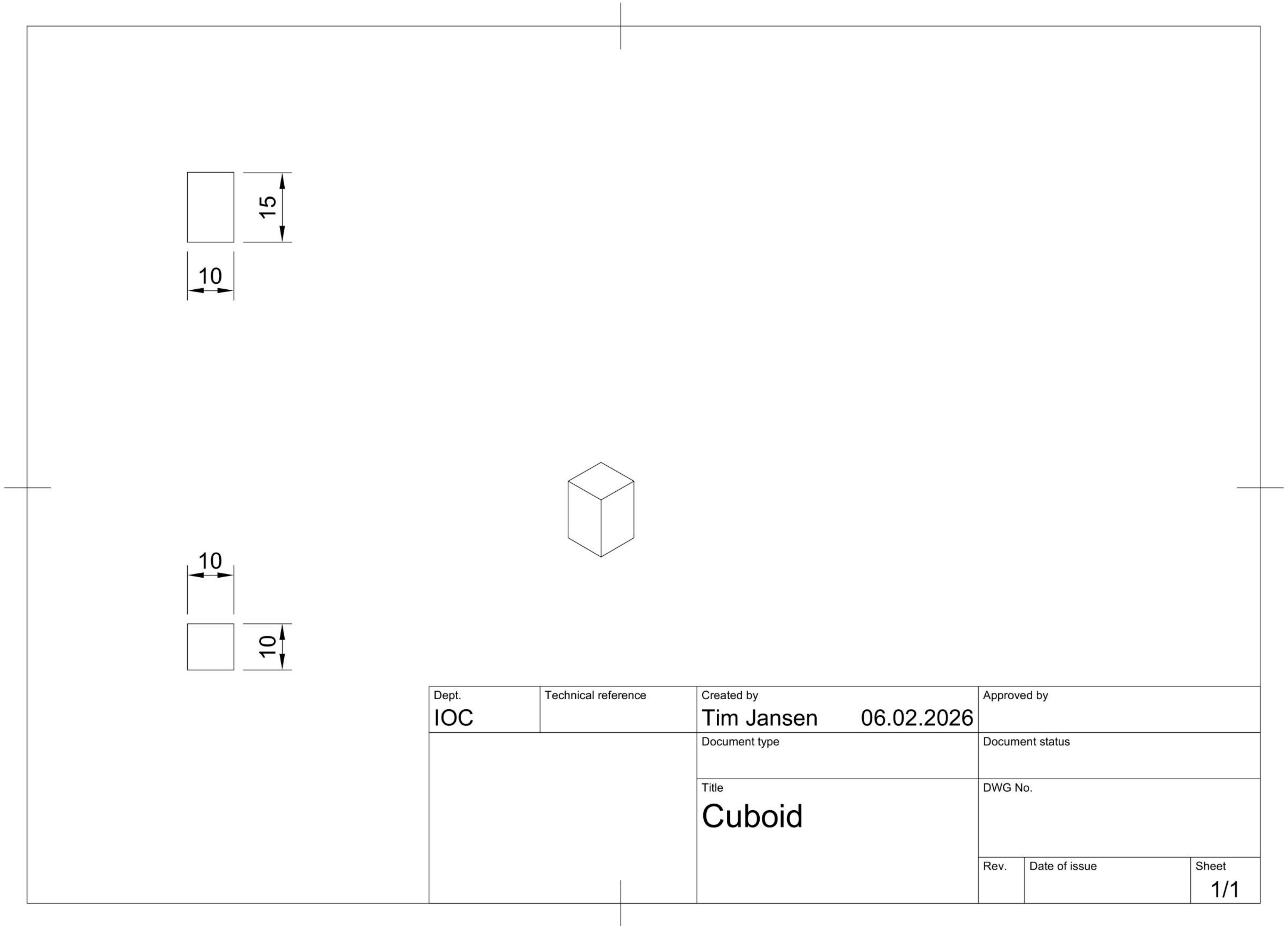
Cube 10mm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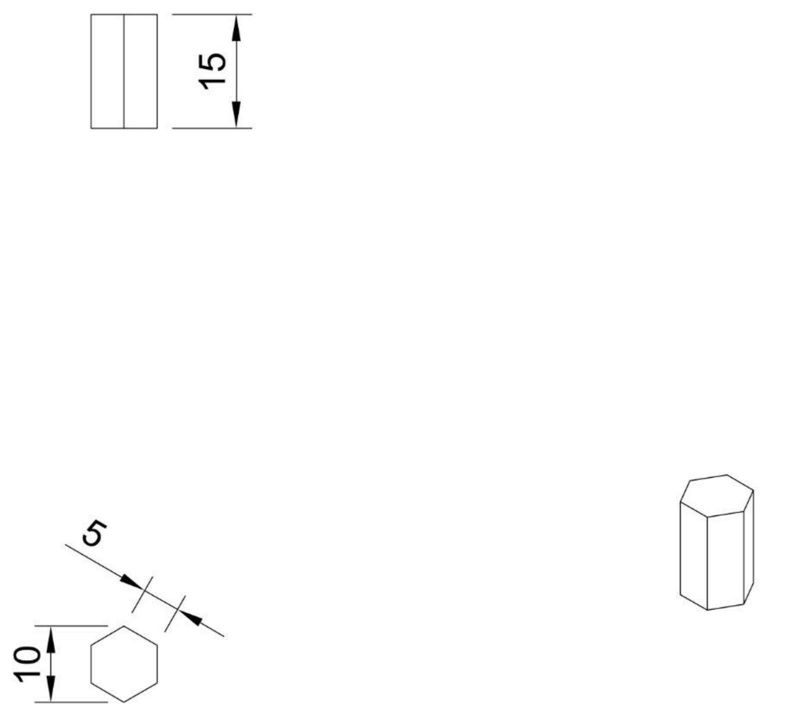
Cuboid



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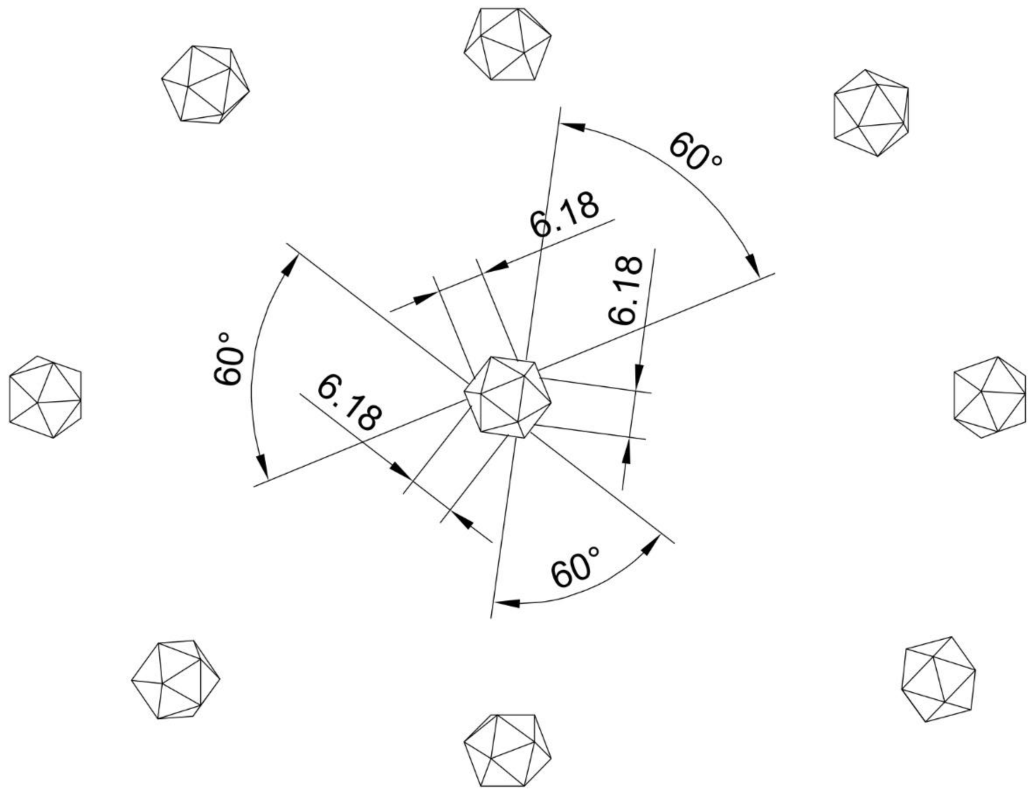
Hexagonal Prism



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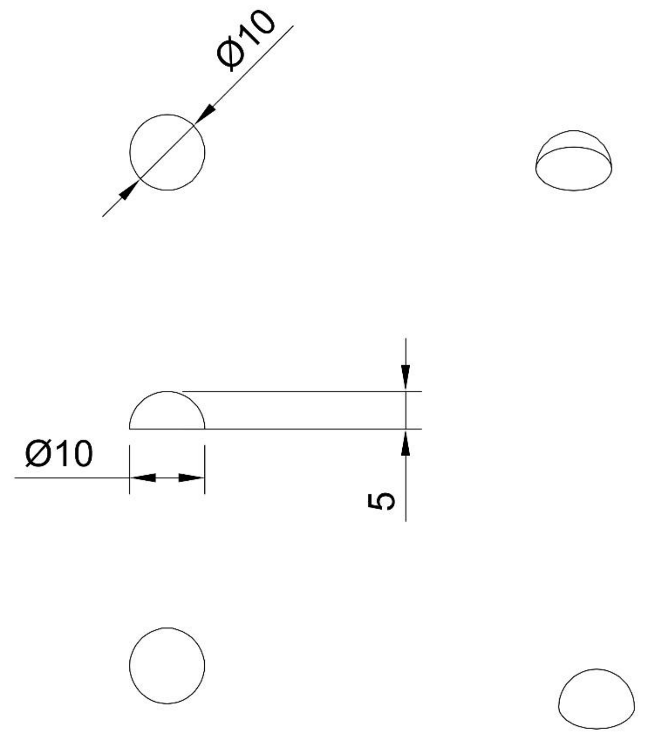
Icosahedron



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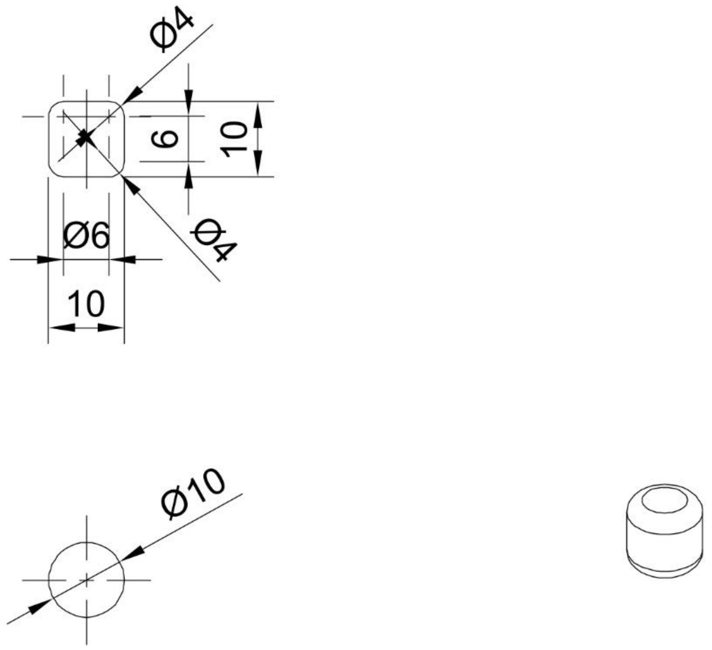
Half Sphere



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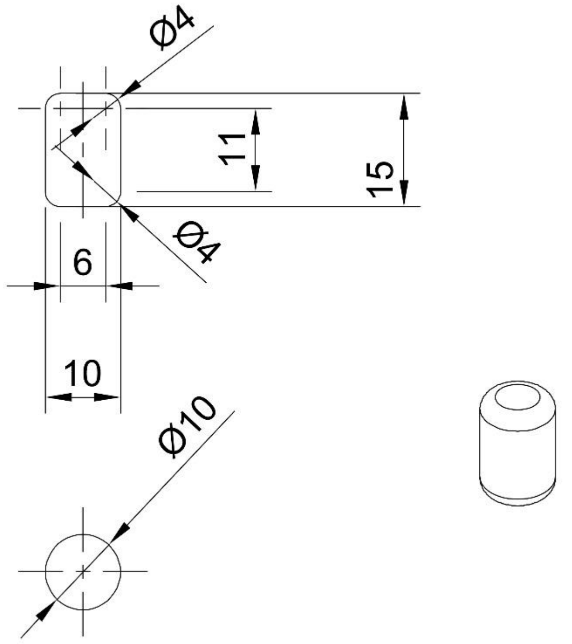
Cylinder h10-d10



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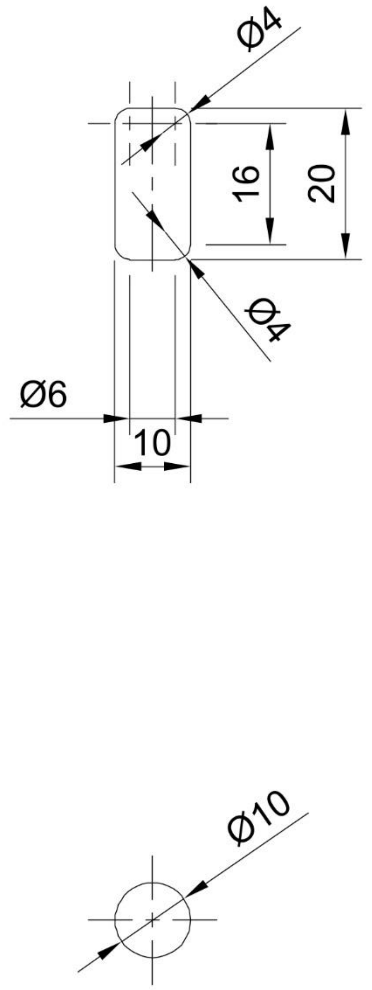

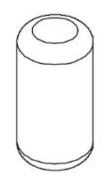

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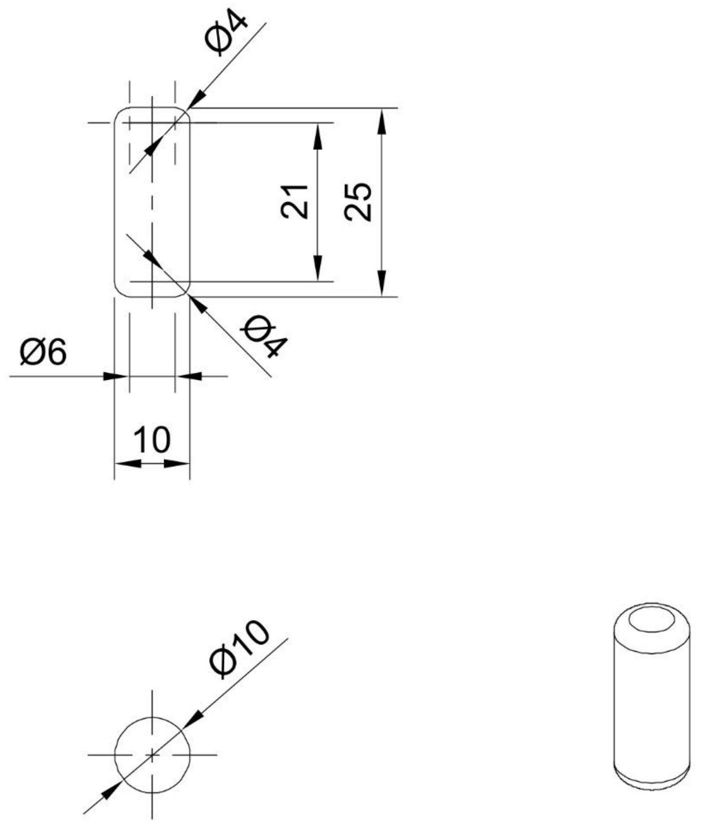
Cylinder h20-d10



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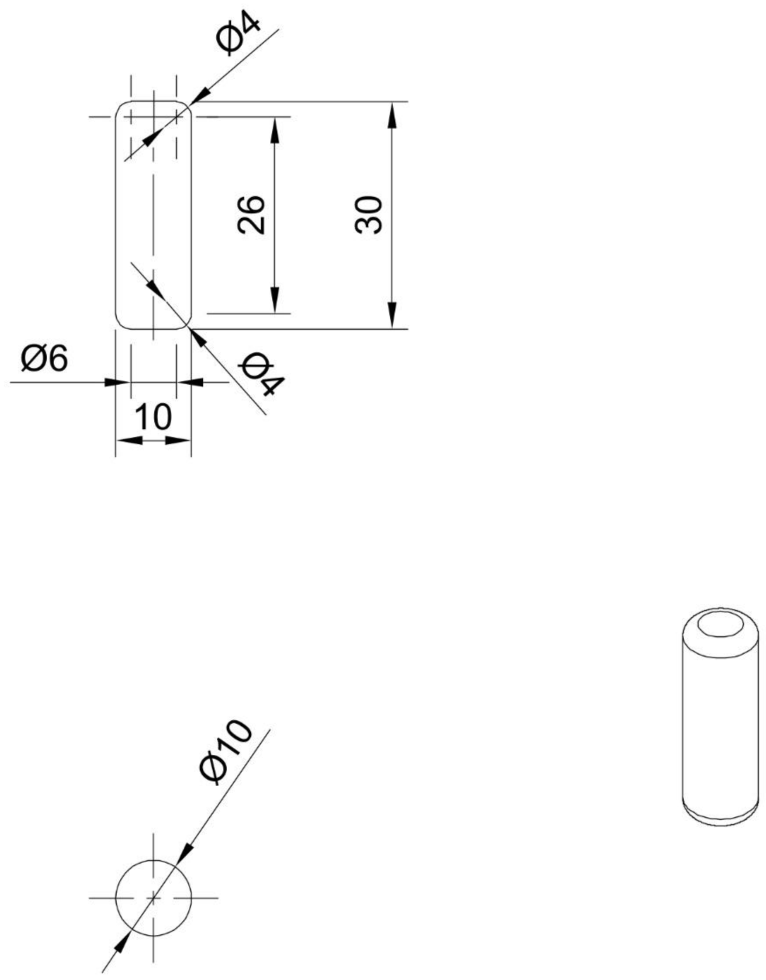
Cylinder h25-d10



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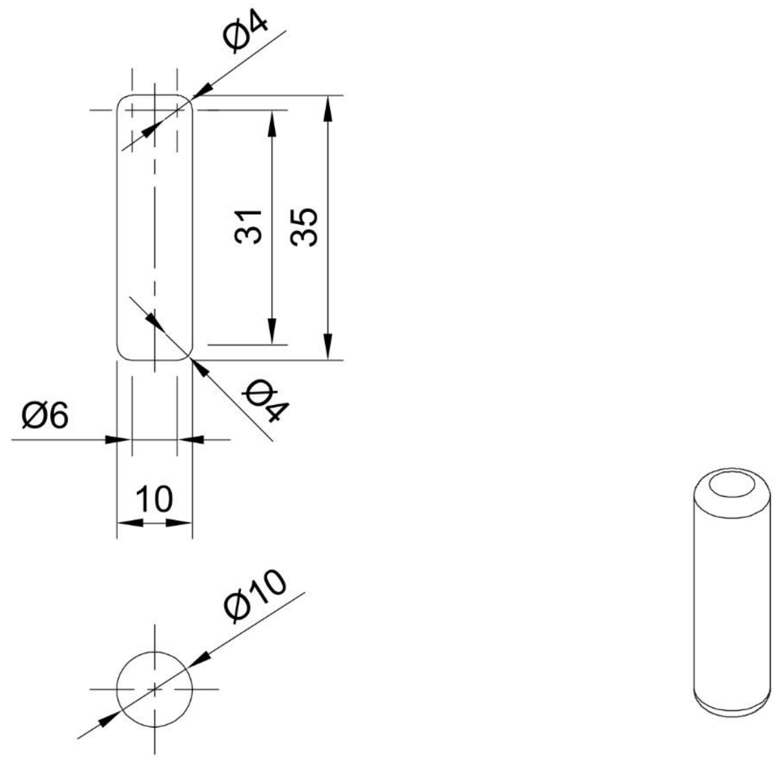
Cylinder h30-d10



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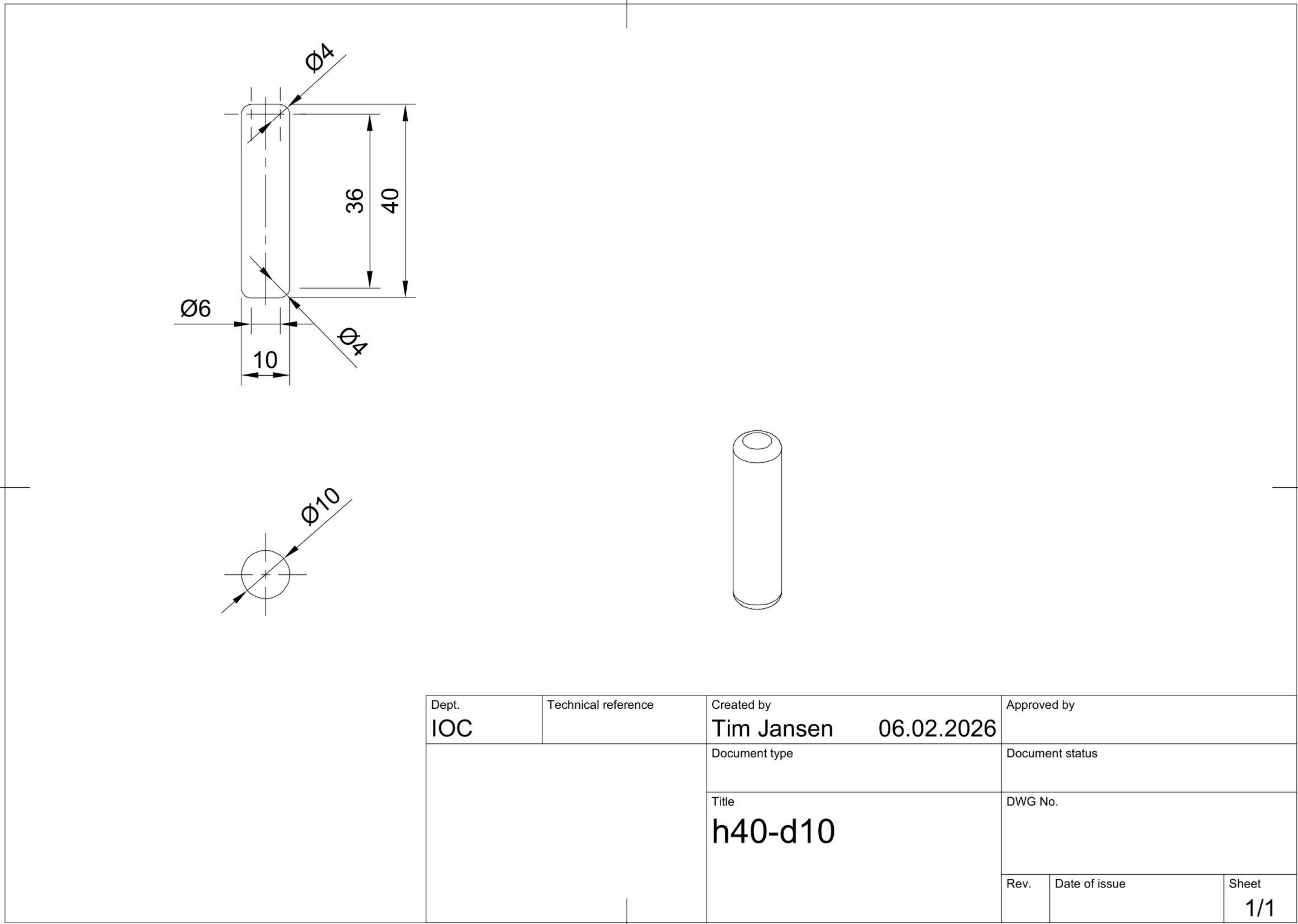
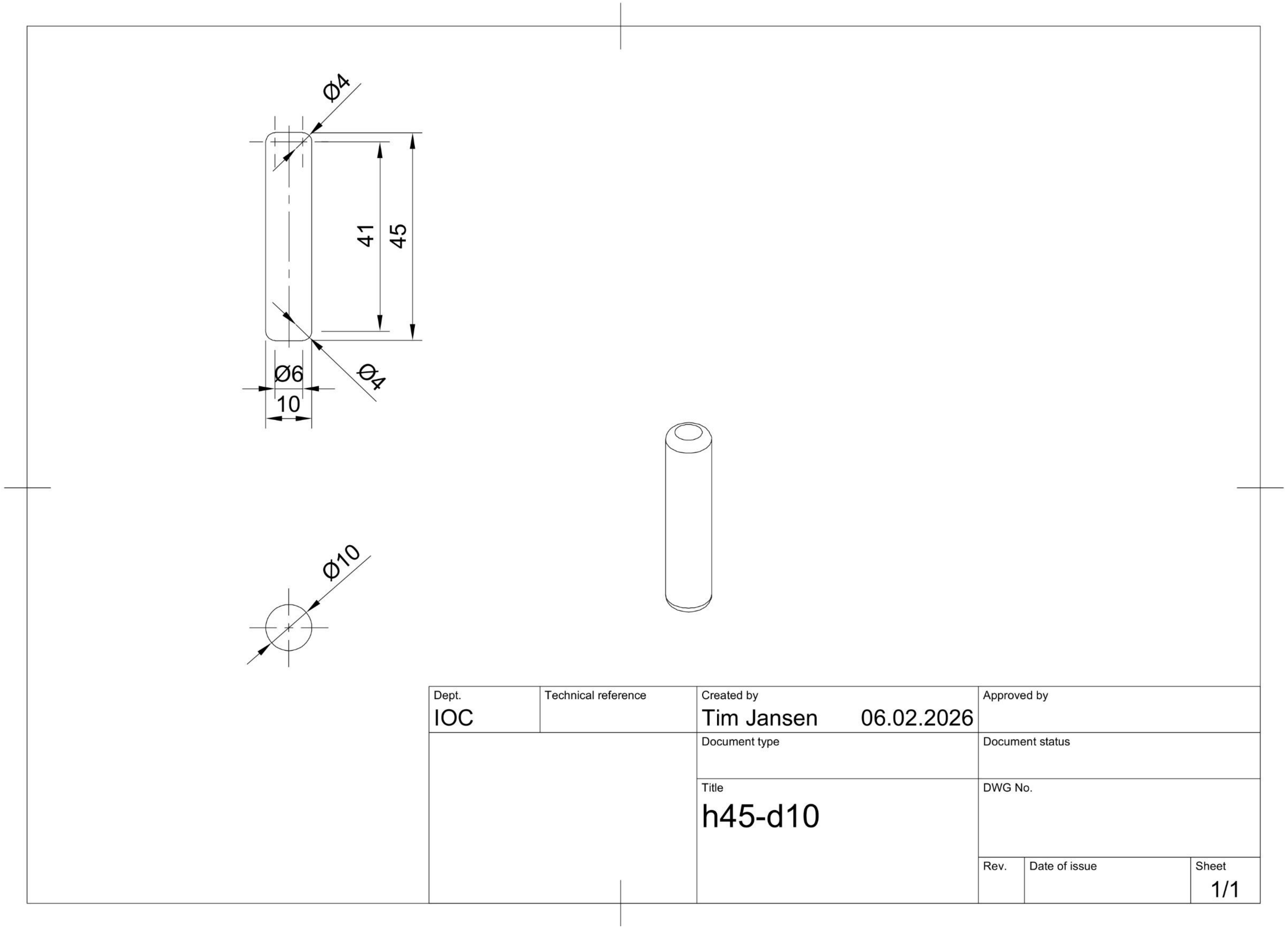
Cylinder h35-d10



Cylinder h40-d10



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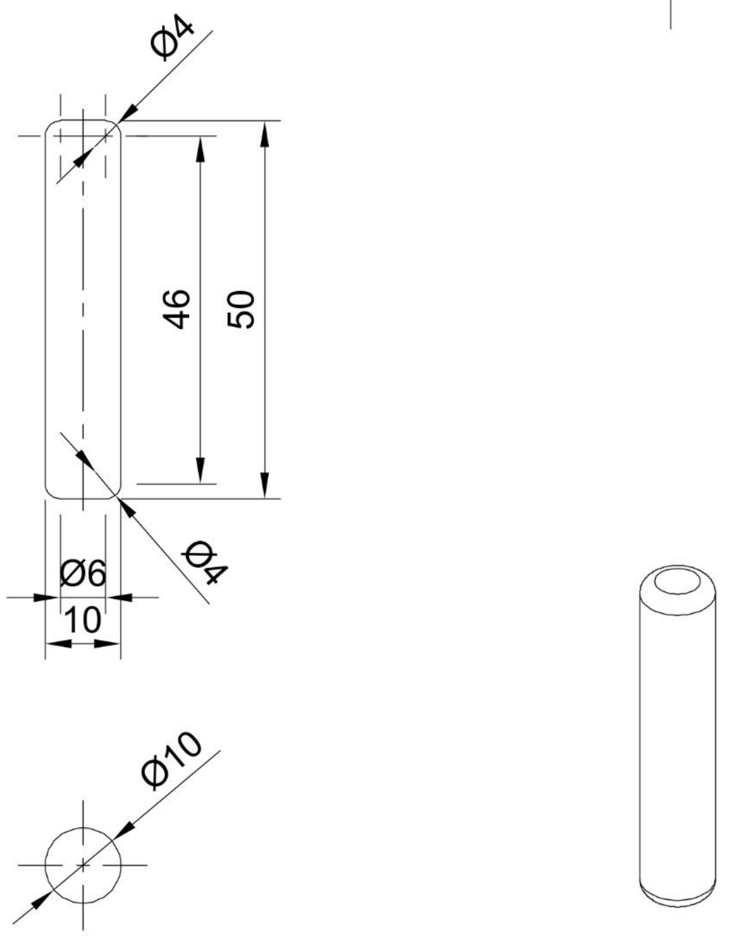

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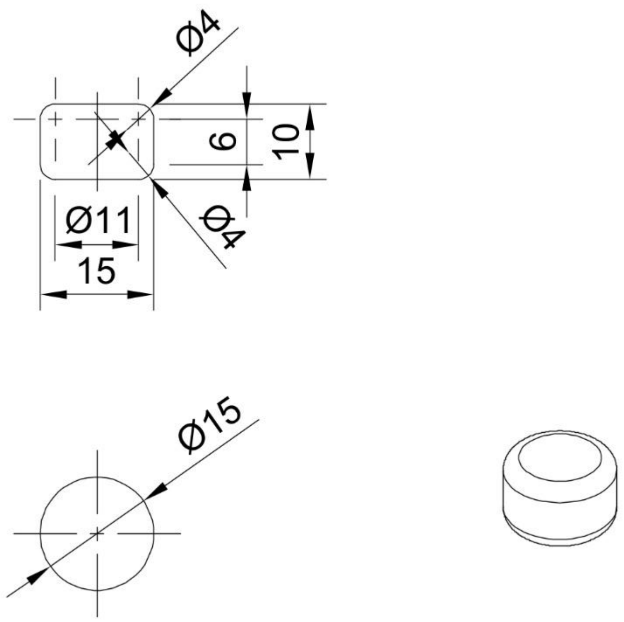
Cylinder h50-d10



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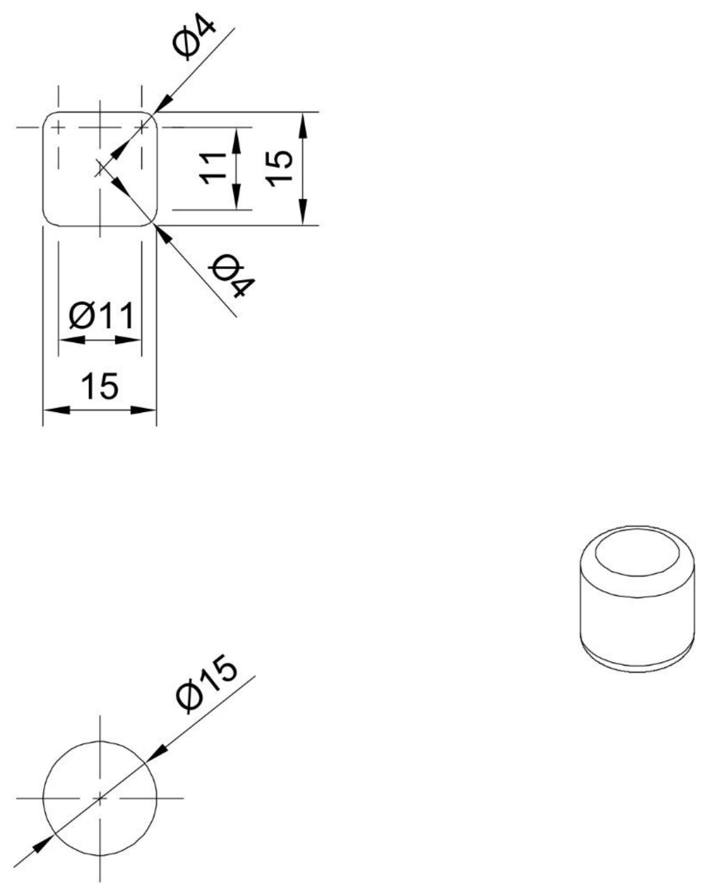
Cylinder h10-d15



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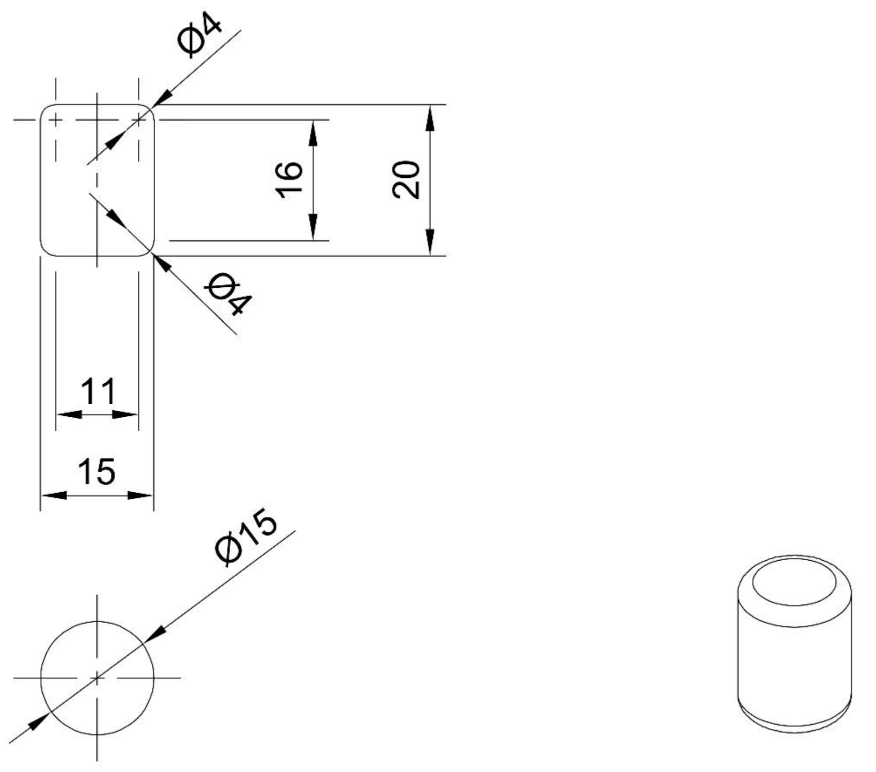
Cylinder h15-d15



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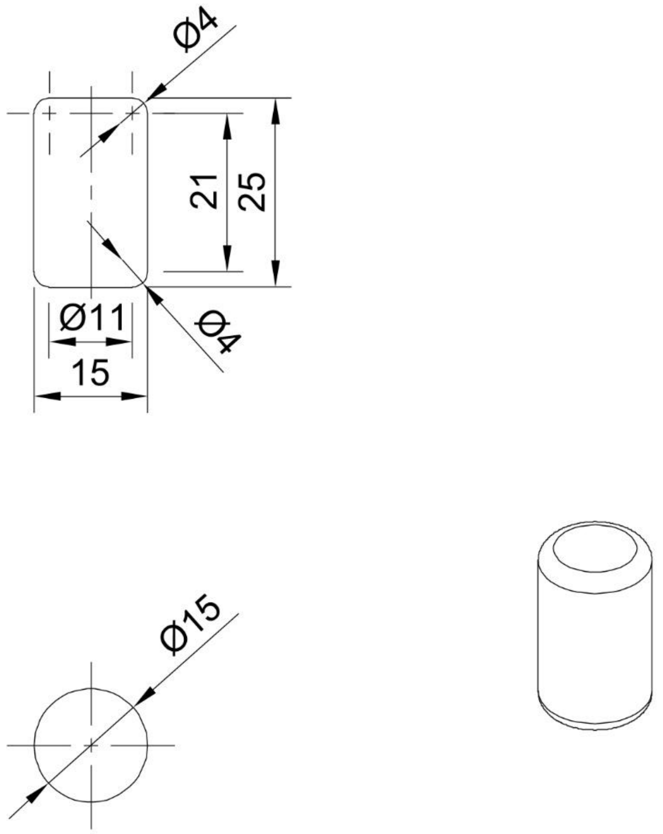
Cylinder h20-d15



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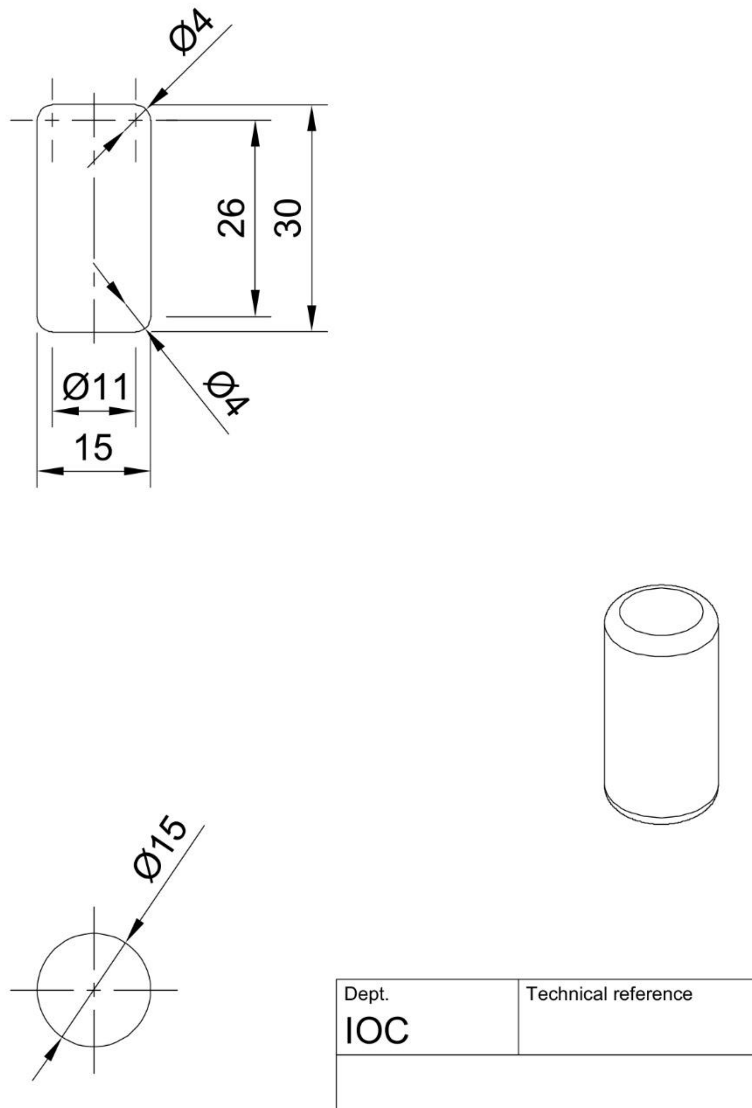
Cylinder h25-d15



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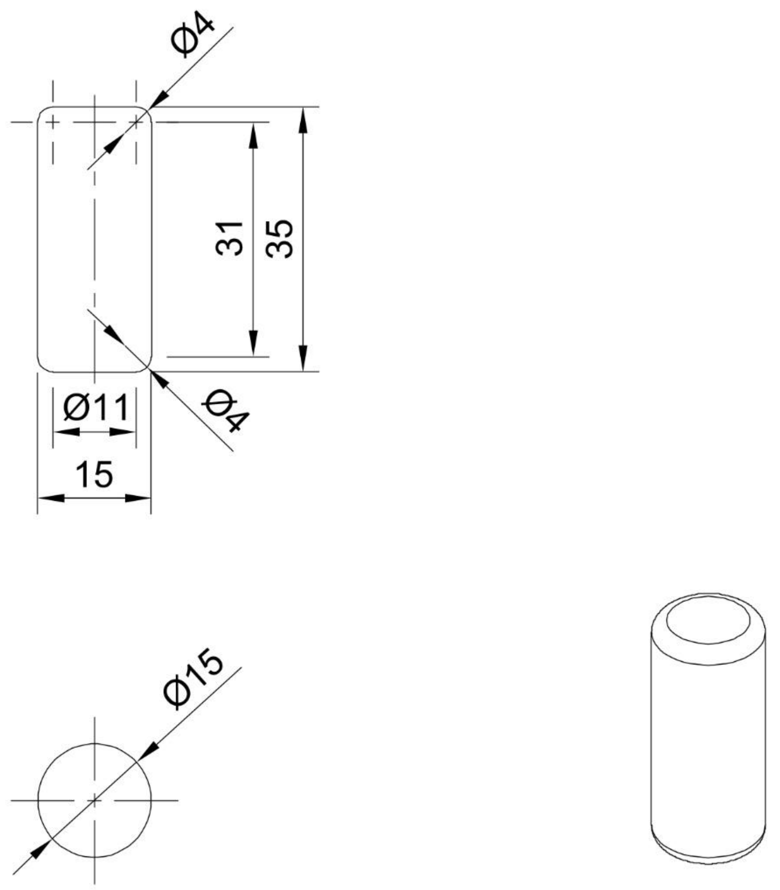
Cylinder h30-d15



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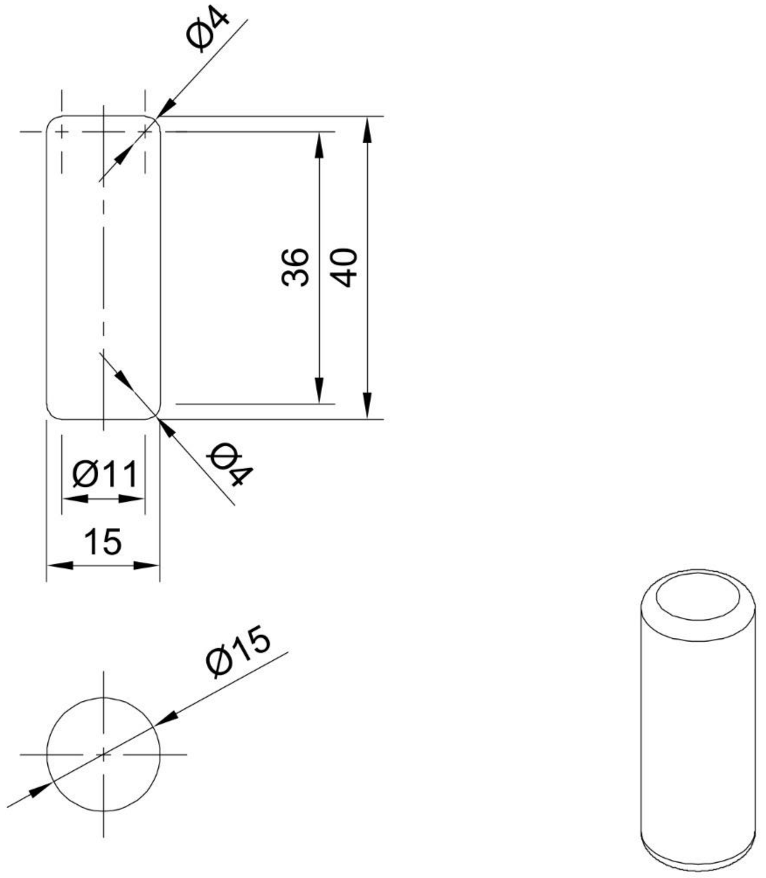
Cylinder h35-d15



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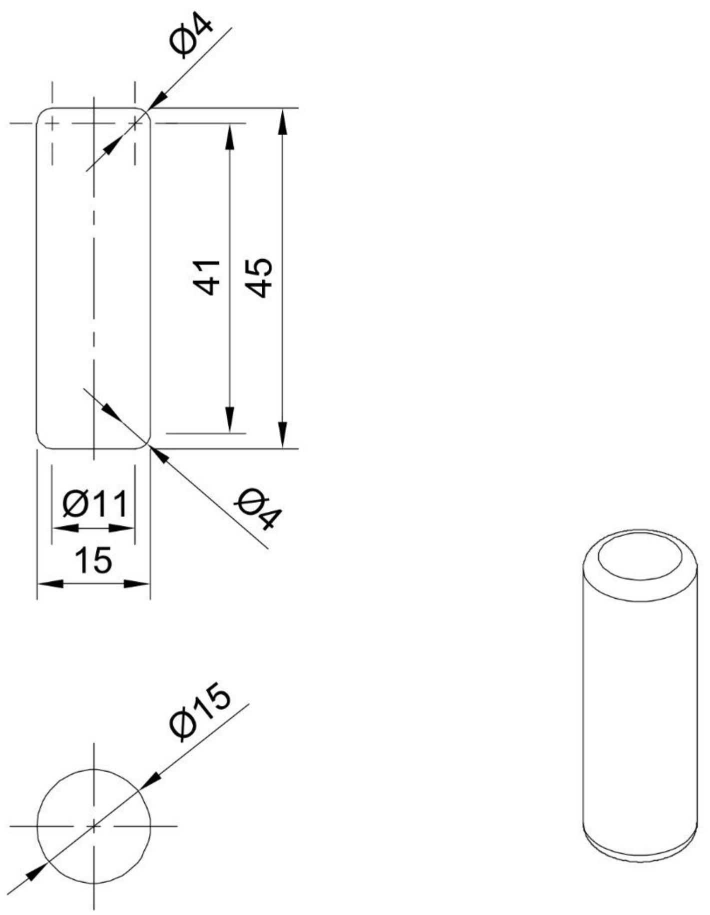
Cylinder h40-d15



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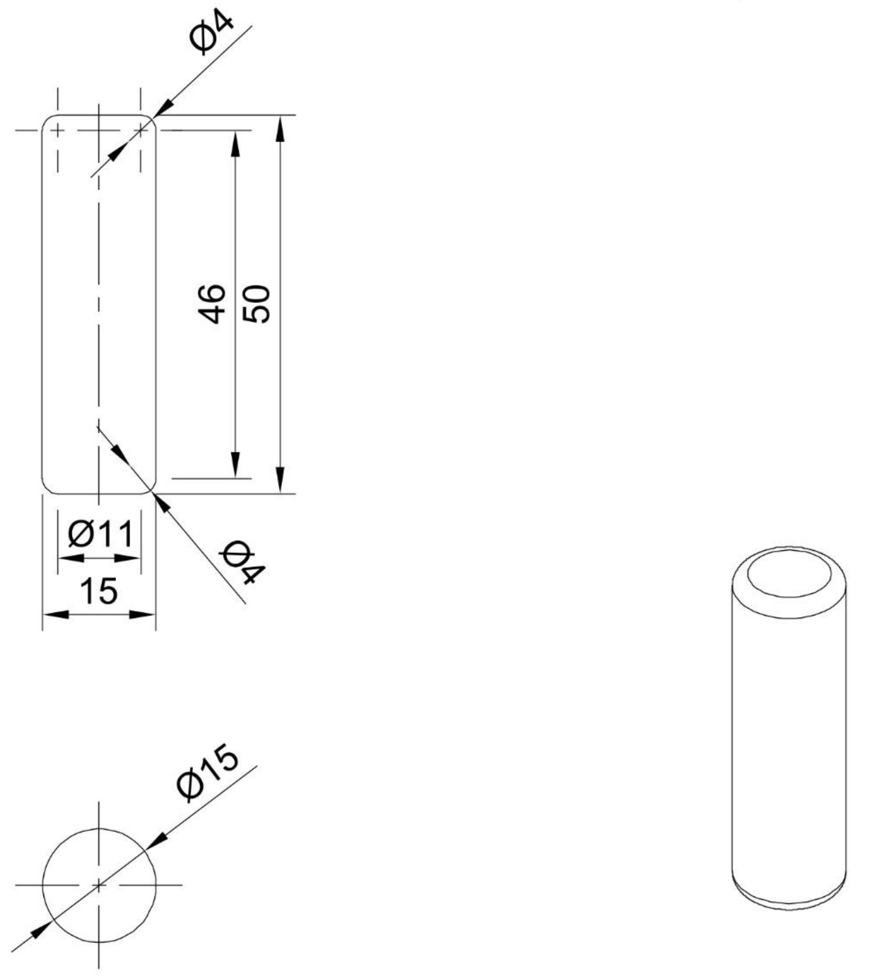
Cylinder h45-d15



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Cylinder h50-d15

2 3D printing of the designs

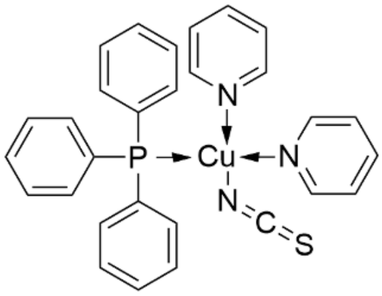
All milling vessels and milling bodies were printed on an Anycubic Photon Mono M5s Pro SLA-Printer. The used resins are Anycubic High Clear Resin for the milling vessels and Tough Resin 2.0 clear as well as Tough Resin 2.0 gray for the milling Bodies. According to the safety data sheet, the High Clear Resin consists of: oxybis(methyl-2,1-ethanediyl) diacrylate, poly(oxy-1,2-ethanediyl), α,α'-[(1-methylethylidene)di-4,1-phenylene]bis[ω-[(1-oxo-2-propen-1-yl)oxy], (octahydro-4,7-methano-1*H*-indenediyl)bis(methylene) diacrylate and bisphenol A polyetheenglycol diether dimethacrylate. The Tough Resin 2.0 consists of: 2-oxepanone, homopolymer, 2-[(1-oxo-2-propen-1-yl)oxy]ethyl ester, isodecyl methacrylate, 1,2-ethanediyl bisacrylate, phenyl-bis-(2,4,6-trimethylbenzoyl)-phosphine oxide. This safety data sheet information of Anycubic was not validated further. The 3D models were translated into print files with the software CHITUBOX. The printing settings were adjusted to the resins rather than just using the settings from Anycubic. The exact settings are given in Table S1.

Table S 1 3D printing settings for the different resin types.

	High Clear Resin		Tough Resin 2.0	
Layer Height	0.05 mm		0.05 mm	
Bottom Layer Count	3		5	
Exposure Time	3.6 s		3.6 s	
Bottom Exposure Time	22 s		22 s	
Transition Layer Count	6		3	
Transition Type	Linear		Linear	
Light-off Delay	0.5 s		1 s	
Bottom Lifting Distance	4 mm	4 mm	6 mm	4 mm
Lifting Distance	4 mm	4 mm	6 mm	4 mm
Bottom Lift Speed	360 mm/min	360 mm/min	360 mm/min	360 mm/min
Lifting Speed	360 mm/min	360 mm/min	360 mm/min	500 mm/min
Bottom Retract Speed	360 mm/min	360 mm/min	360 mm/min	360 mm/min
Retract Speed	360 mm/min	360 mm/min	360 mm/min	500 mm/min

After printing, the residual resin was removed with isopropanol and acetone, the parts were dried under airflow, and the prepared parts were cured under UV-light inside of the Anycubic curing station. The wavelength of the UV-light was 405 nm, and the time was three minutes.

3 Synthesis of copper(I) thiocyanate bipyridine triphenylphosphine



Copper(I) thiocyanate bipyridine triphenylphosphine was synthesized as described in the literature.[S1] Copper(I)thiocyanate (2.99 g, 16.3 mmol, 1 equiv.) and triphenylphosphine (4.31 g, 16.3 mmol, 1.00 equiv.) were dissolved in pyridine (50.0 mL, 618 mmol), and the mixture was heated to 70 °C for 3 hours. Then, pyridine was removed by distillation to obtain the solid complex. The solid complex was recrystallized from pyridine (15 mL), and the recrystallized solid was washed with ice cold pyridine (10 mL). The crystalline complex was dried under reduced pressure and stored under air at room temperature.

^{1}H-NMR (600 MHz, $CDCl_3$): δ = 8.73 (s, 4H, $H_{Arom.}$), 7.70 (t, J = 7.6 Hz, 2H, $H_{Arom.}$), 7.39 – 7.30 (m, 12H, $H_{Arom.}$), 7.30 – 7.23 (m, 7H, $H_{Arom.}$).

^{13}C NMR (151 MHz, $CDCl_3$): δ = 136.13 (SCN), 133.67 (d, J = 14.9 Hz, $C_{Arom.}$), 132.91 (d, J = 30.7 Hz, $C_{Arom.}$), 129.66 ($C_{Arom.}$), 128.52 (d, J = 9.3 Hz, $C_{Arom.}$), 123.93 ($C_{Arom.}$).

4 Imaging procedure and hit map creation

All experiments were performed using the left arm of the shaker mill IST636 of the company Insolido. A Samsung Galaxy S20FE was used for the imaging procedure. Long exposure photography was done in the photography pro mode, with an ISO 50 and two seconds exposure time. High speed footage was obtained by using the super slow-mo setting of the mobile phone. The frame rate for this setting is 960 fps over a duration of 0.5 seconds. The illumination with red light was done by a LingsFire headlamp. For the videography the 3D-printed milling jar was filled with 100 mg of the copper complex and the 3D-printed milling body. The milling vessel was fixed in the left arm of the shaker mill, the flashlight was placed on the lid, and the mobile phone was placed in front of the mill with a tripod. The mill was started, and the video was taken at the moment when it reached the desired frequency. When the video was captured, the mill was stopped and the procedure was repeated. After two captured videos, the copper complex was renewed, and another two videos were taken. After a total of six videos, the measurement was finished and a total of three seconds of milling time were captured. The videos were transferred to a computer, and each frame was analyzed regarding hits in the normal as well as the mirror image. The hits were marked in an excel sheet with a grid pattern resembling the vessel geometry. Each time another hit occurred at the same spot the number of this grid part was increased by one. The extension of hits was considered by affecting more parcels at once so that the energy input was taken into account.

5 Hit maps and hit counts of the milling body geometries

The hit maps are in the following order:

Milling Ball, Cube 5 mm, Cube 10 mm, Cuboid, Hexagonal Prism, Icosahedron, Half Sphere, Cylinders 10 mm diameter, Cylinders 15 mm diameter.

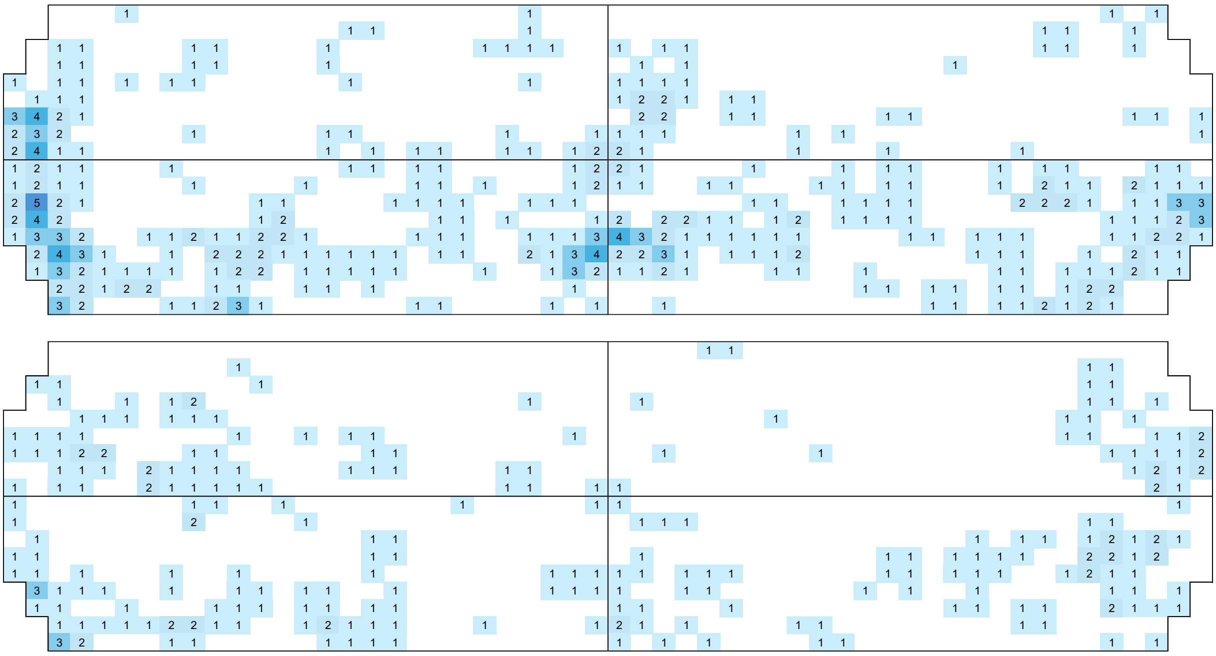


Figure S 1 Hit maps of the reference milling ball at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure.

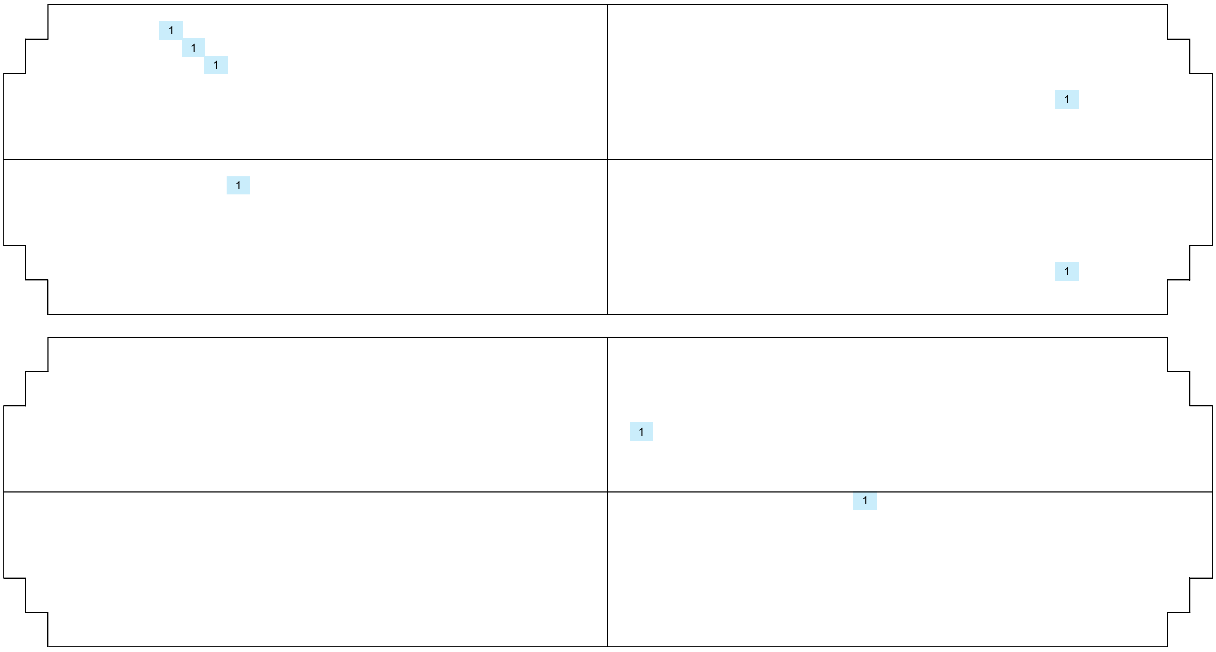


Figure S 2 Hit maps of the 5 mm Cube at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

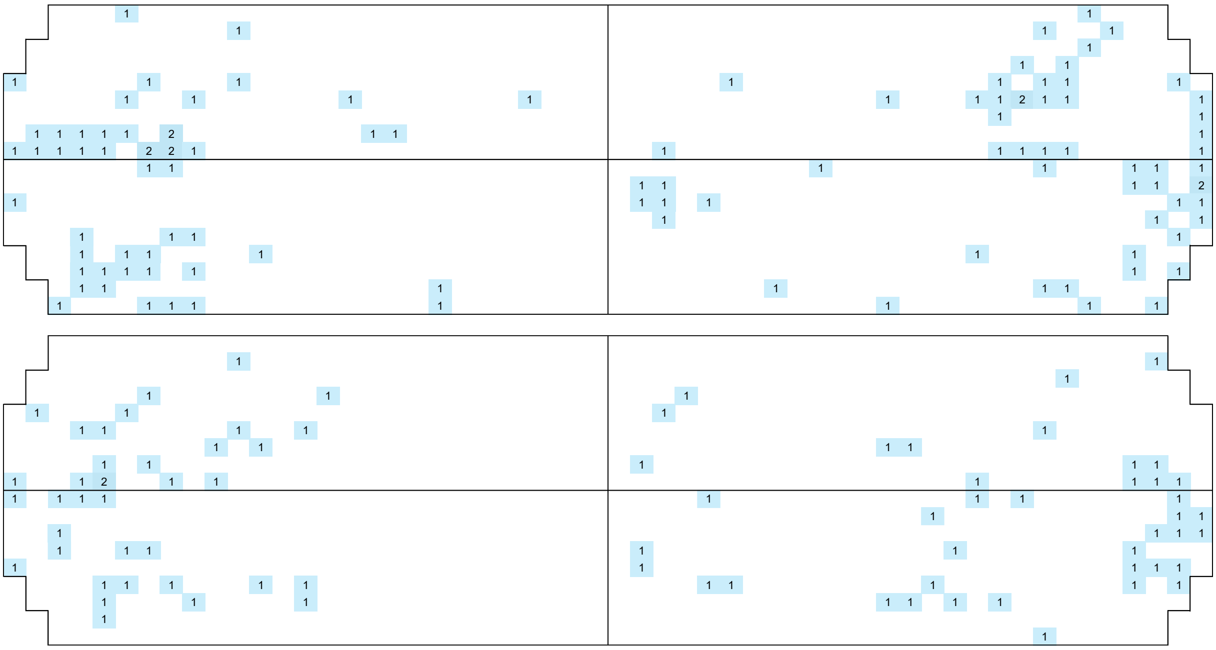


Figure S 3 Hit maps of the 10 mm Cube at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

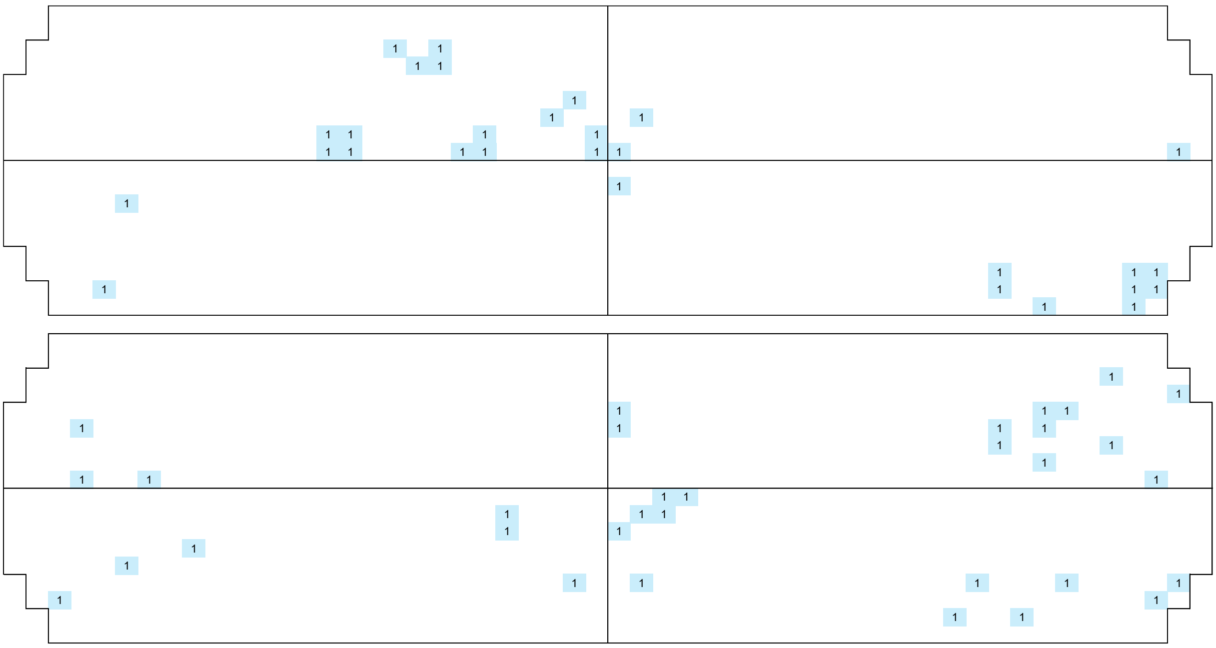


Figure S 4 Hit maps of the Cuboid at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

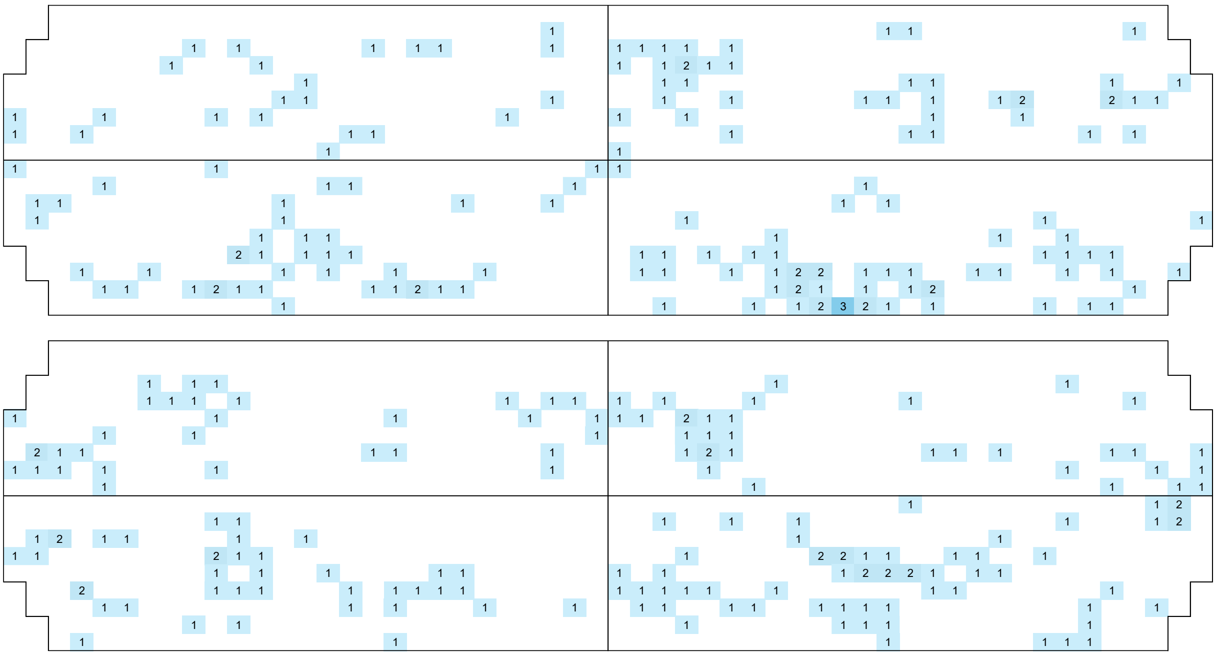


Figure S 5 Hit maps of the Hexagonal Prism at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

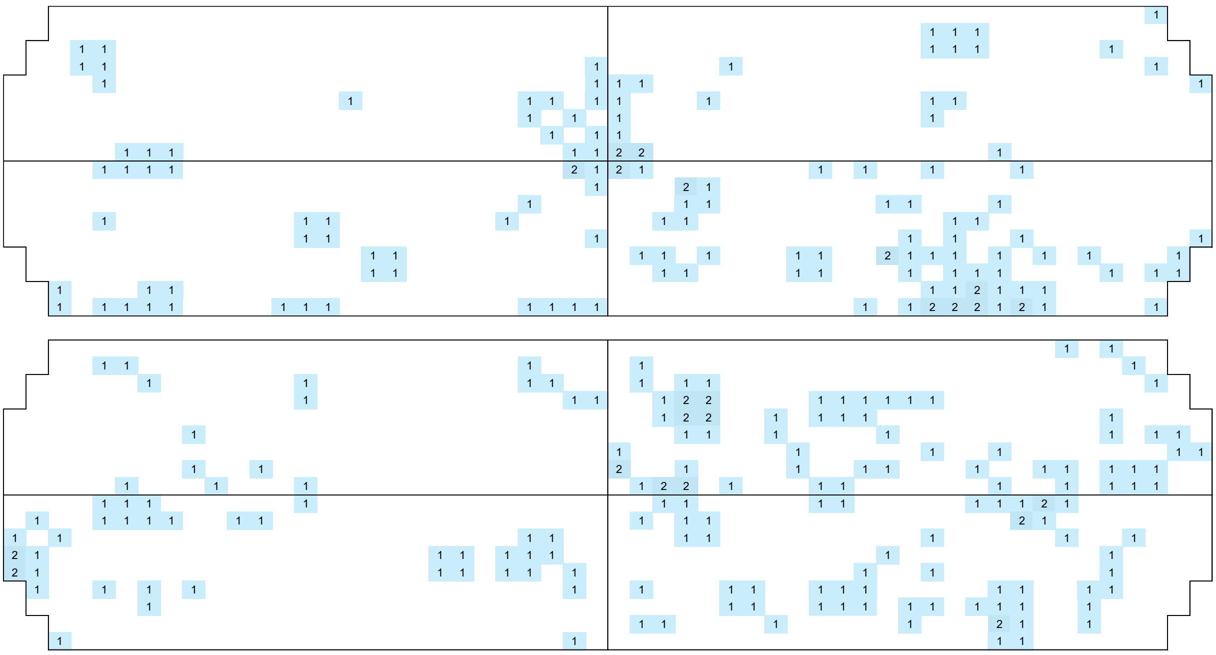


Figure S 6 Hit maps of the Icosahedron at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

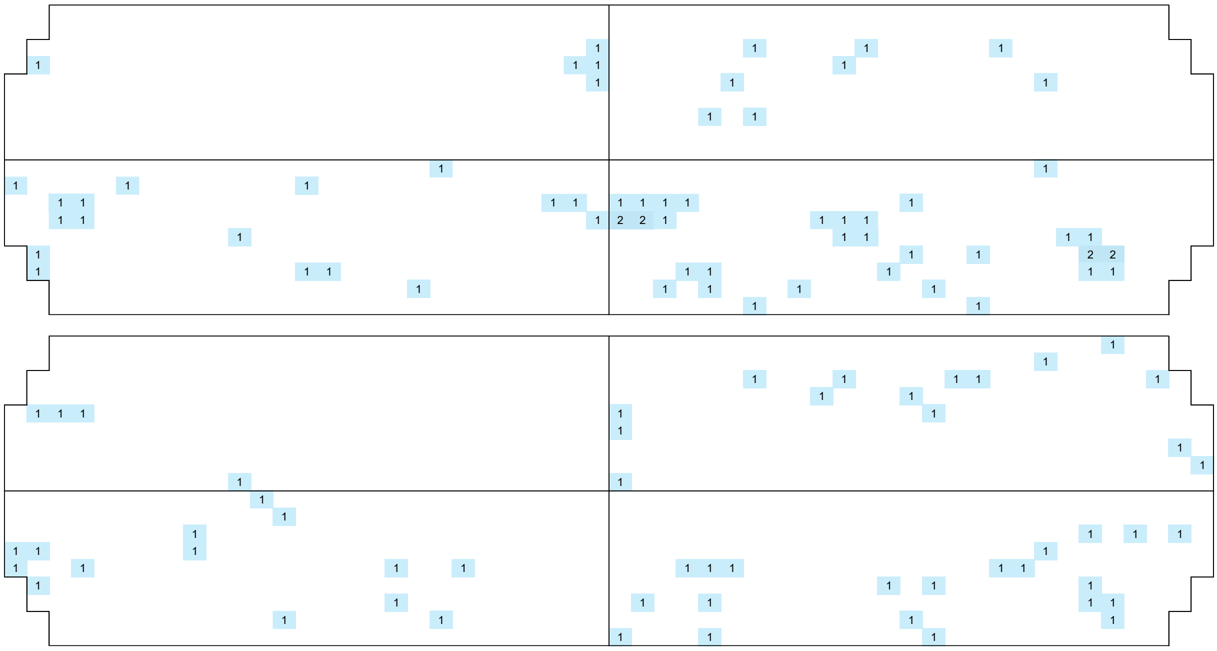


Figure S 7 Hit maps of the Half Sphere at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

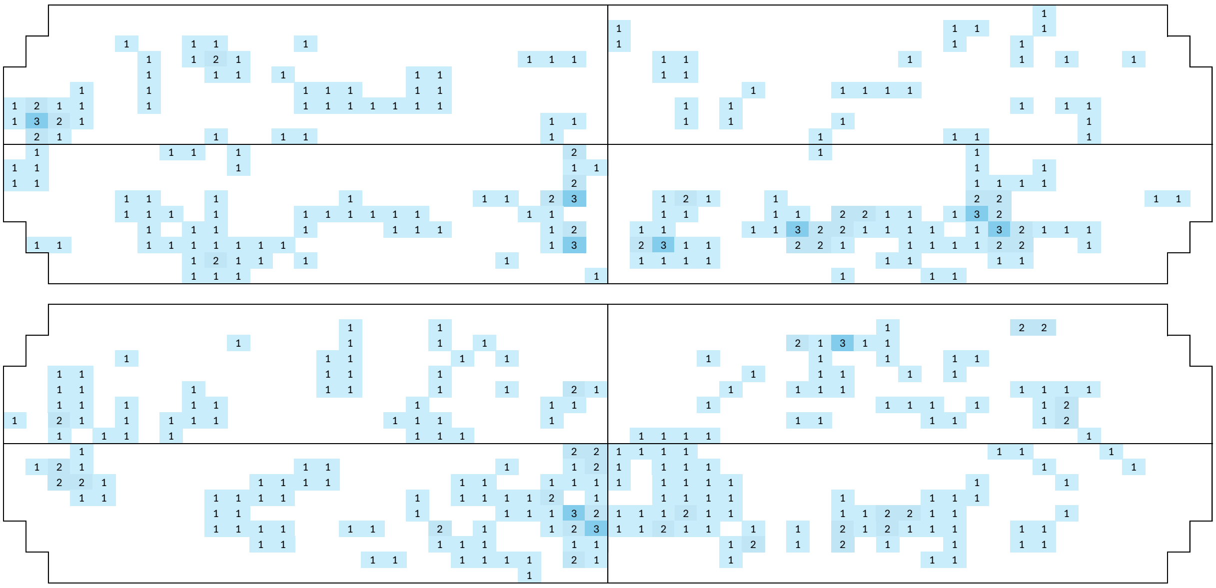


Figure S 8 Hit maps of the h10-d10 Cylinder at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

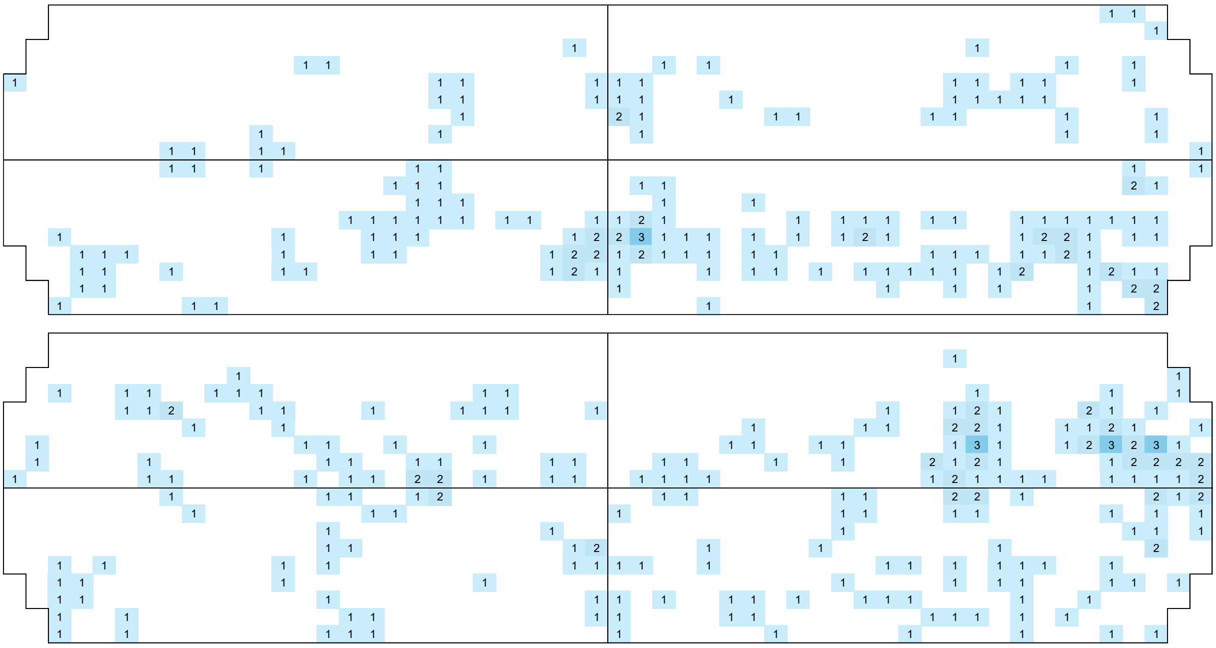


Figure S 9 Hit maps of the h15-d10 Cylinder at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

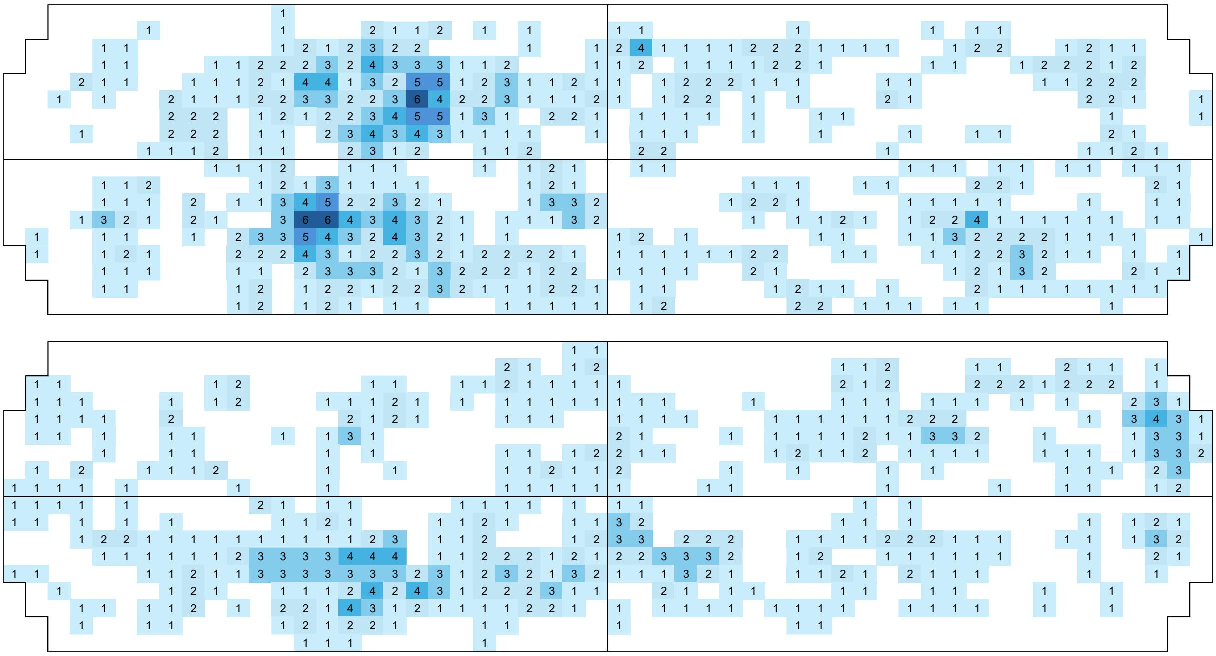


Figure S 10 Hit maps of the h20-d10 Cylinder at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

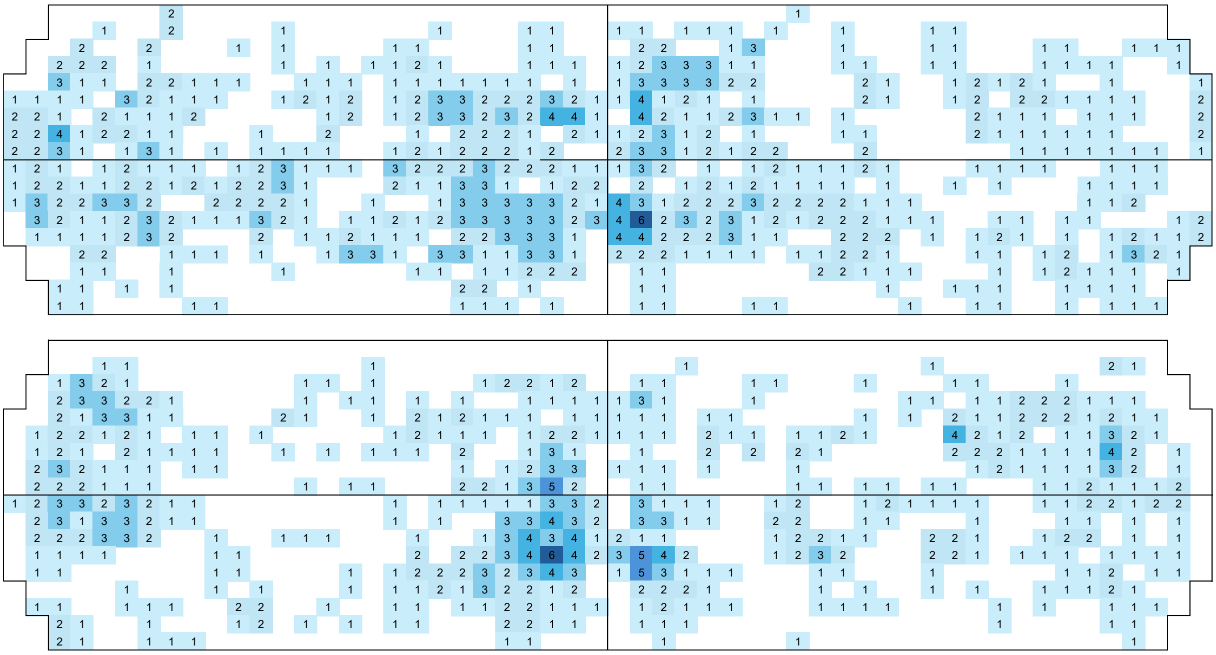


Figure S 11 Hit maps of the h25-d10 Cylinder at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

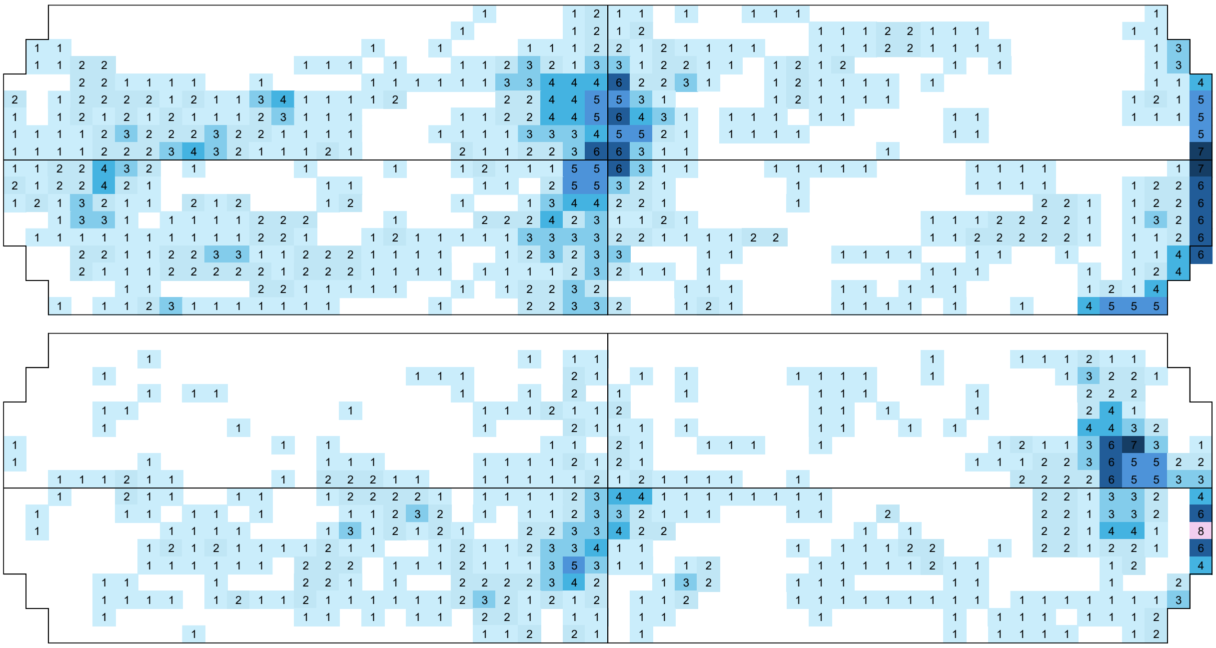


Figure S 12 Hit maps of the h30-d10 Cylinder at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

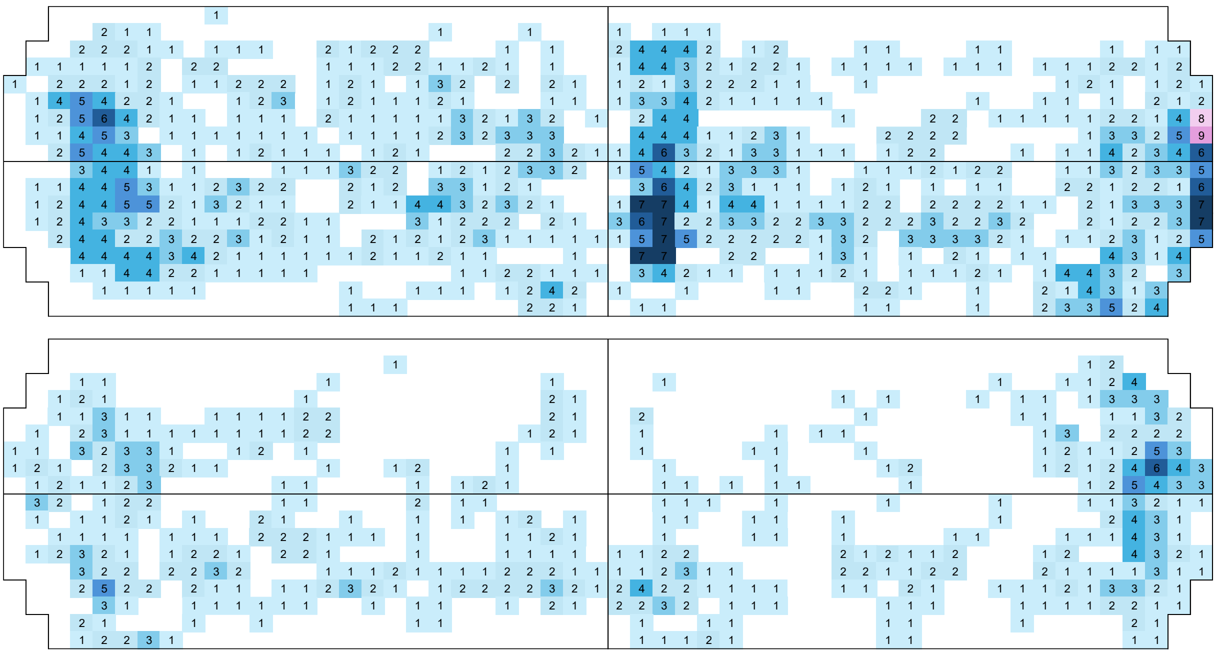


Figure S 13 Hit maps of the h35-d10 Cylinder at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

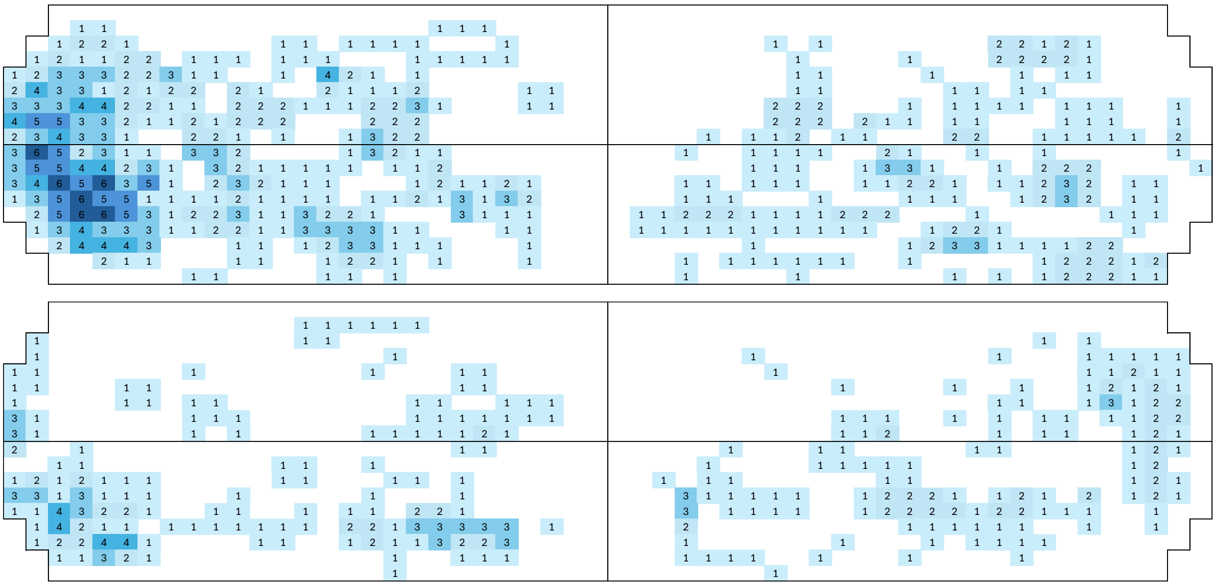


Figure S 14 Hit maps of the h40-d10 Cylinder at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

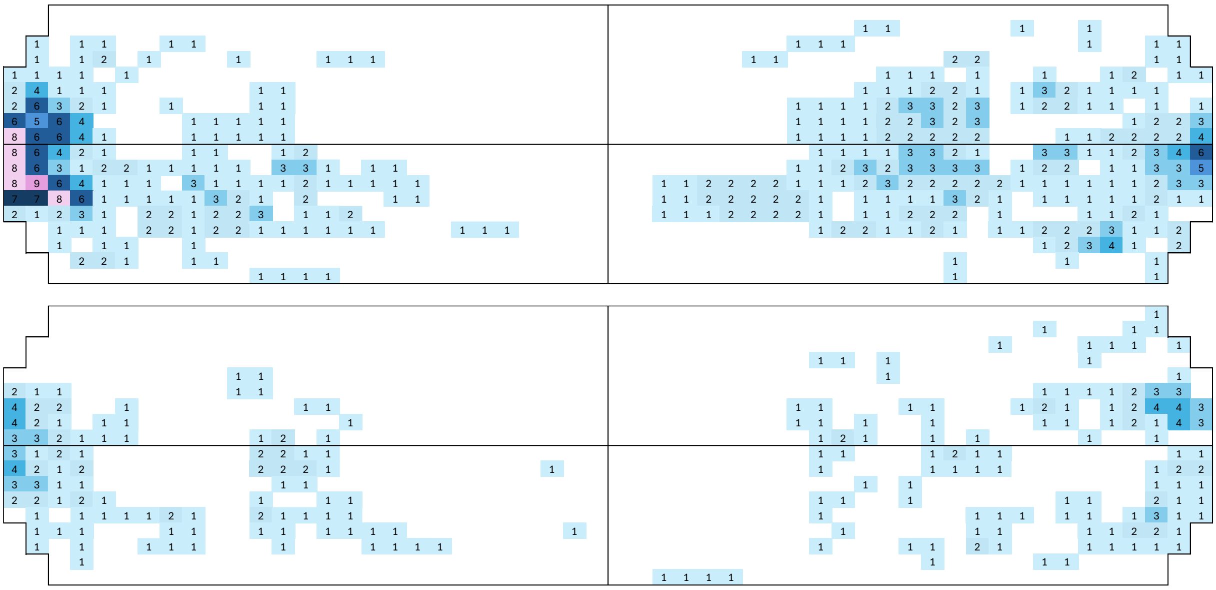


Figure S 15 Hit maps of the h45-d10 Cylinder at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

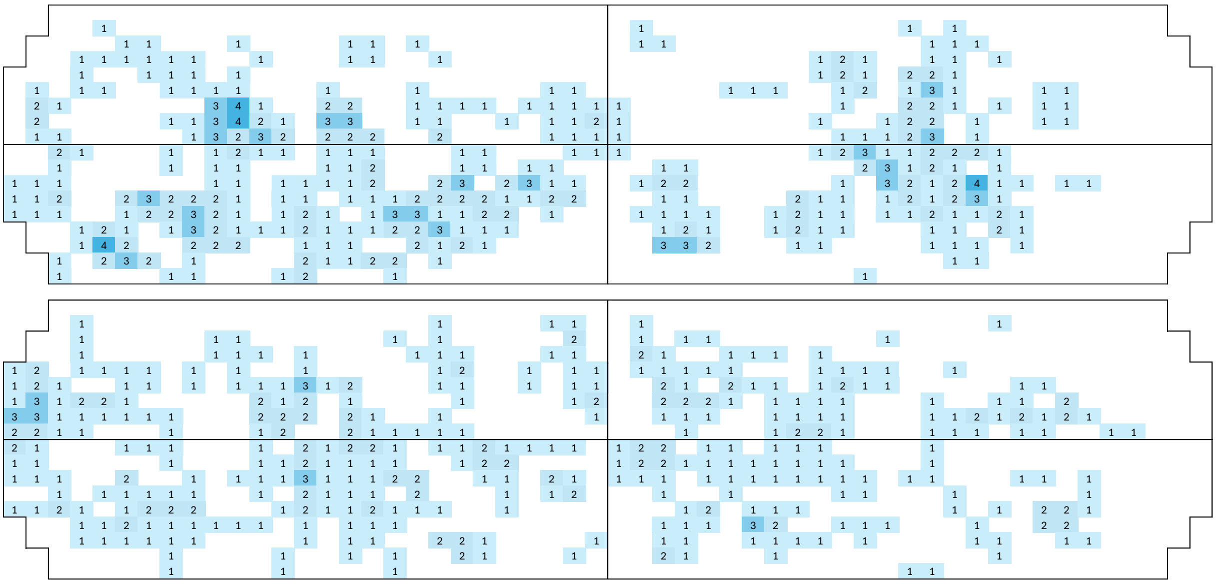


Figure S 16 Hit maps of the h10-d15 Cylinder at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

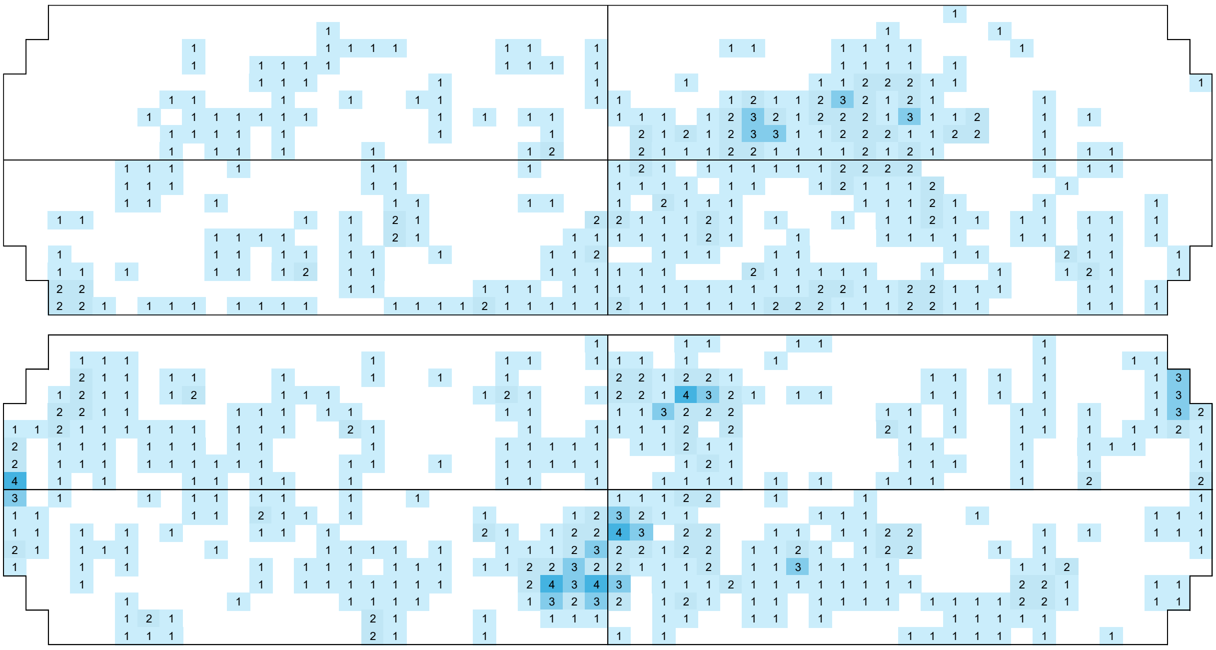


Figure S 17 Hit maps of the h15-d15 Cylinder at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

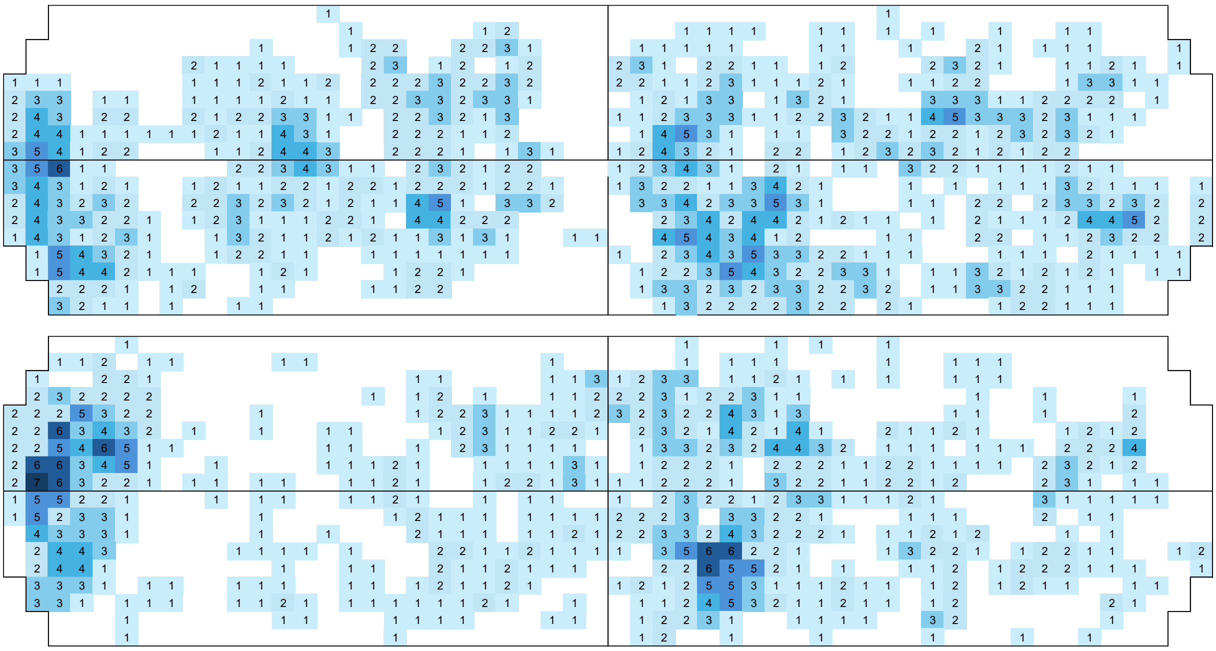


Figure S 18 Hit maps of the h20-d15 Cylinder at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

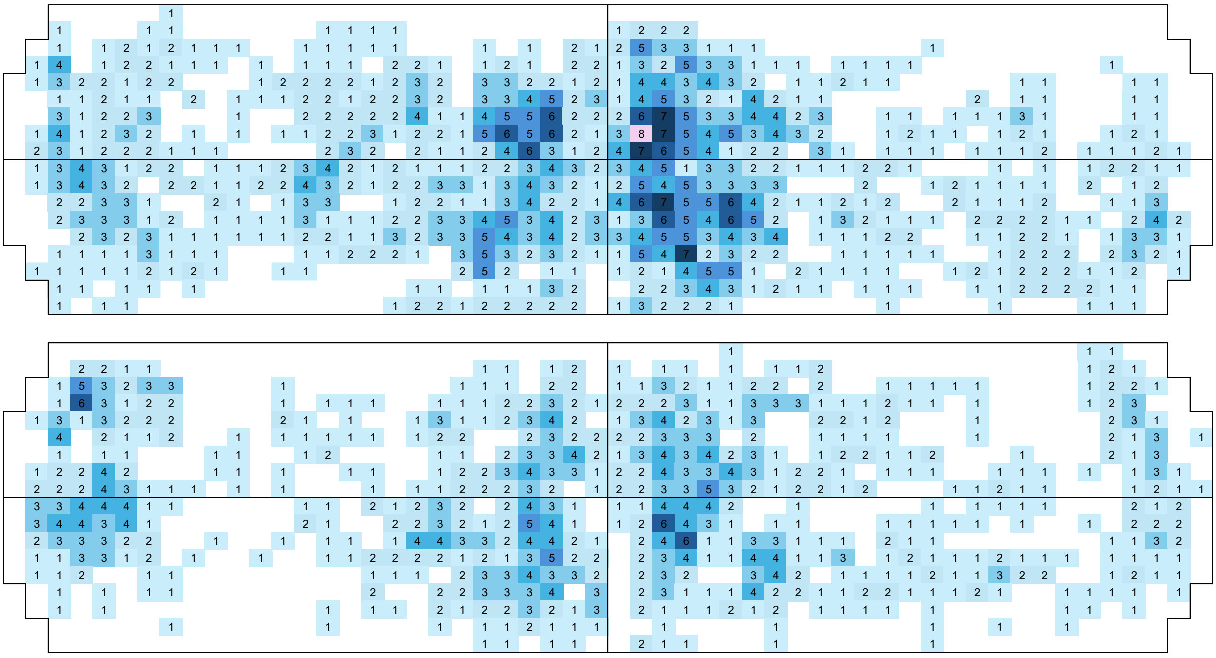


Figure S 19 Hit maps of the h25-d15 Cylinder at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

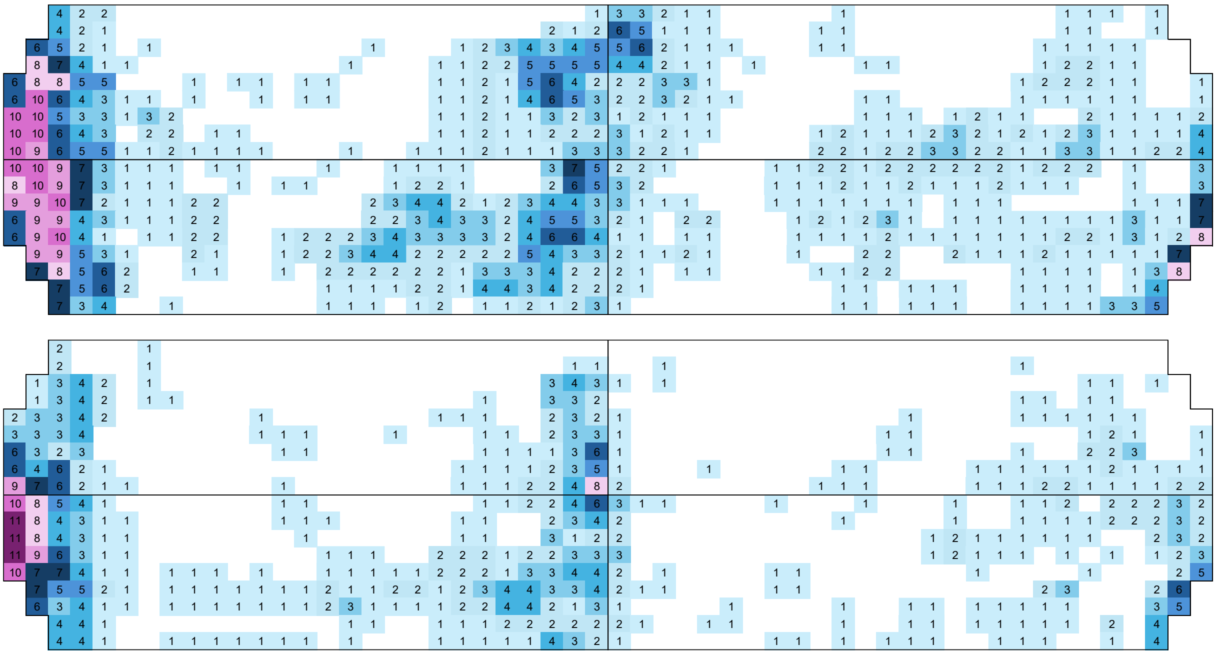


Figure S 20 Hit maps of the h30-d15 Cylinder at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

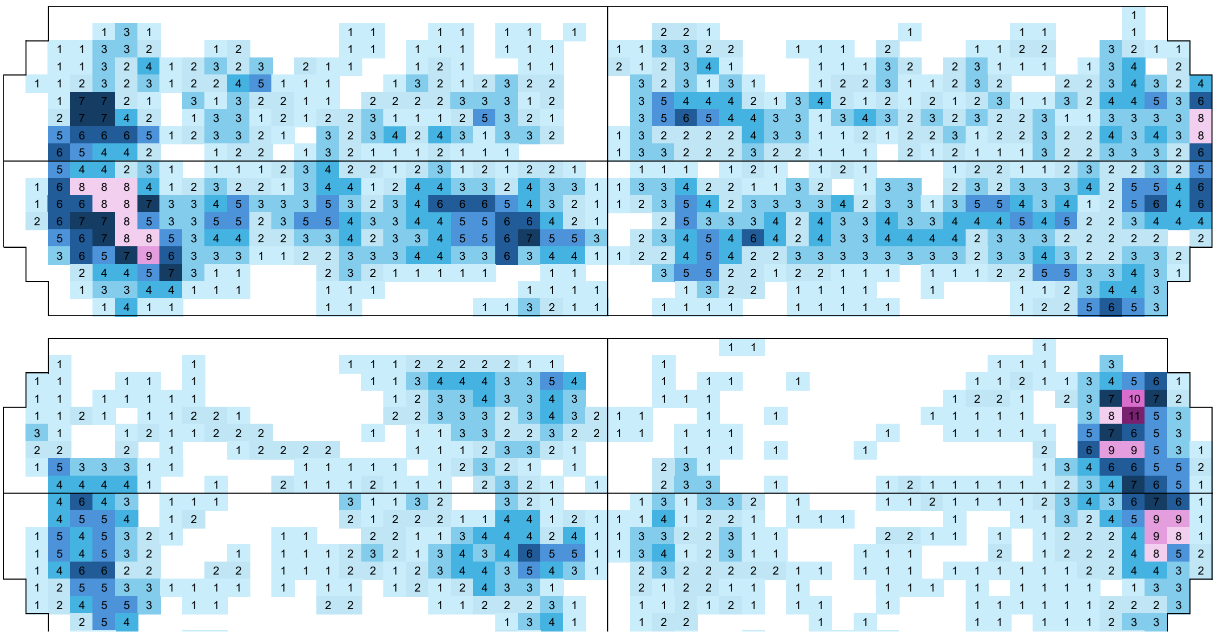


Figure S 21 Hit maps of the h35-d15 Cylinder at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

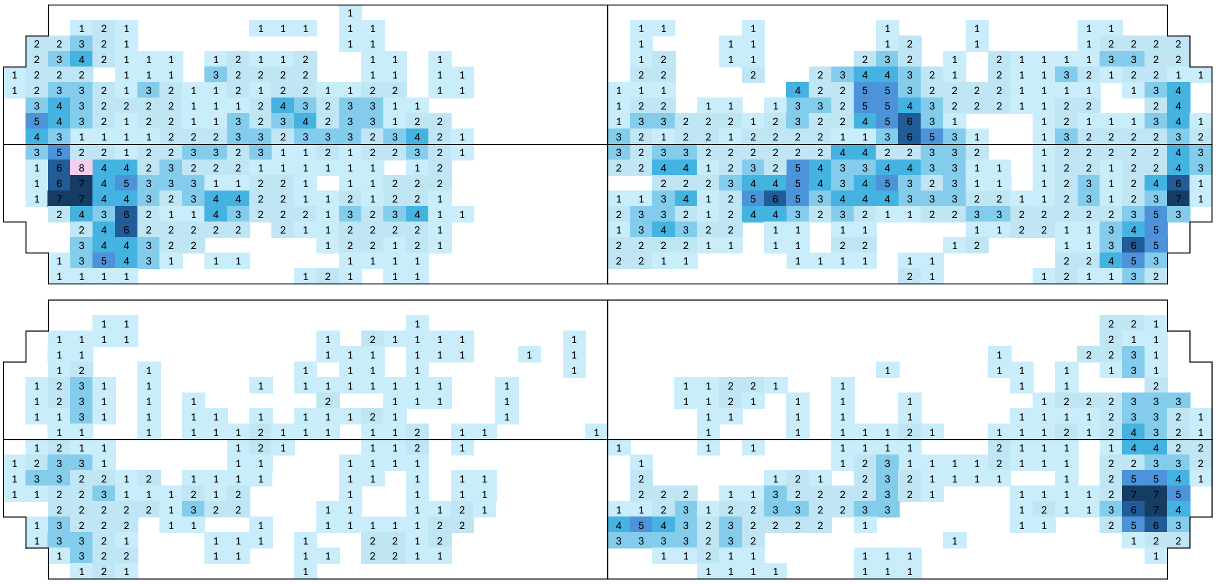


Figure S 22 Hit maps of the h40-d15 Cylinder at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

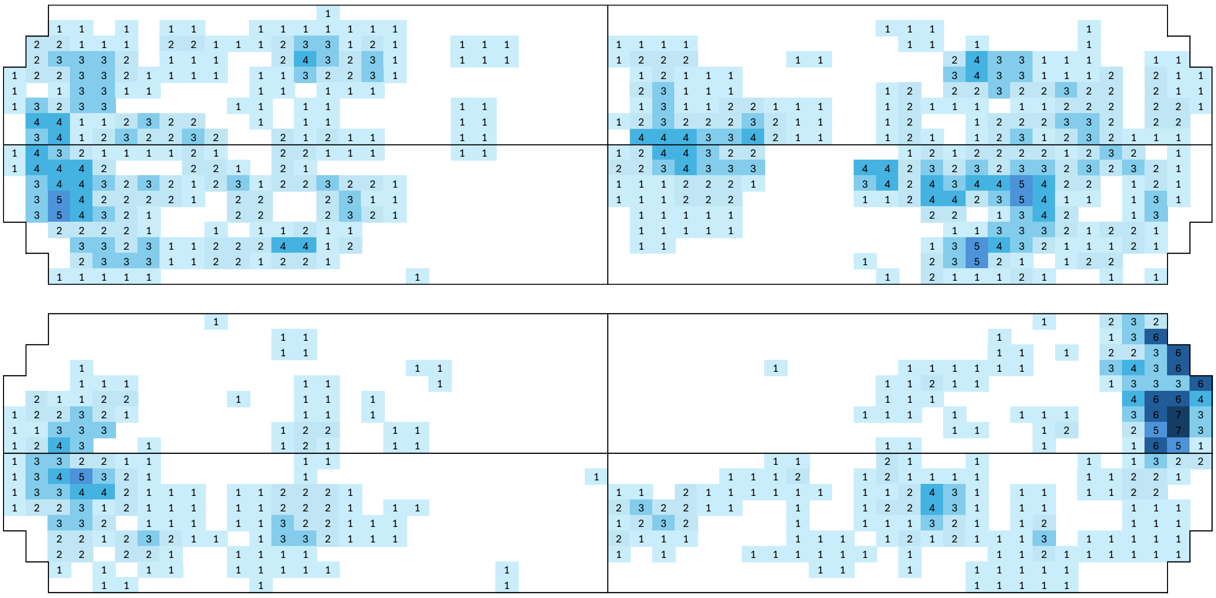


Figure S 23 Hit maps of the h10-d15 Cylinder at a frequency of 25 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

Hit Counts Various Bodies:

Table S 2 Hit counts of the different non cylindrical milling bodies at a frequency of 25 Hz

	Hit Count		
Milling Body	Front View	Bottom View	Overall
Milling Ball	462	264	726
Cube 5mm	6	2	8
Cube 10mm	109	78	187
Cuboid 15x10mm	29	33	62
Hexagonal Prism h15	167	169	336
Icosahedron	148	179	327
Half Sphere	65	54	119
Cylinder h15 d10	201	245	446
Cylinder h15 d15	445	539	984

Hit Counts Cylinders:

Table S 3 Hit Counts of the cylindrical milling bodies at a frequency of 25 Hz

	d10			d15		
Cylinder hight [mm]	Front View	Bottom View	Overall	Front View	Bottom View	Overall
10	251	265	516	482	441	923
15	201	245	446	445	539	984
20	807	626	1433	1165	925	2090
25	864	694	1558	1305	965	2270
30	950	662	1612	1436	846	2282
35	1242	608	1850	1940	1236	3176
40	773	370	1143	1318	656	1974
45	655	279	934	933	583	1516

Frequency Variations for the h30 cylinders:

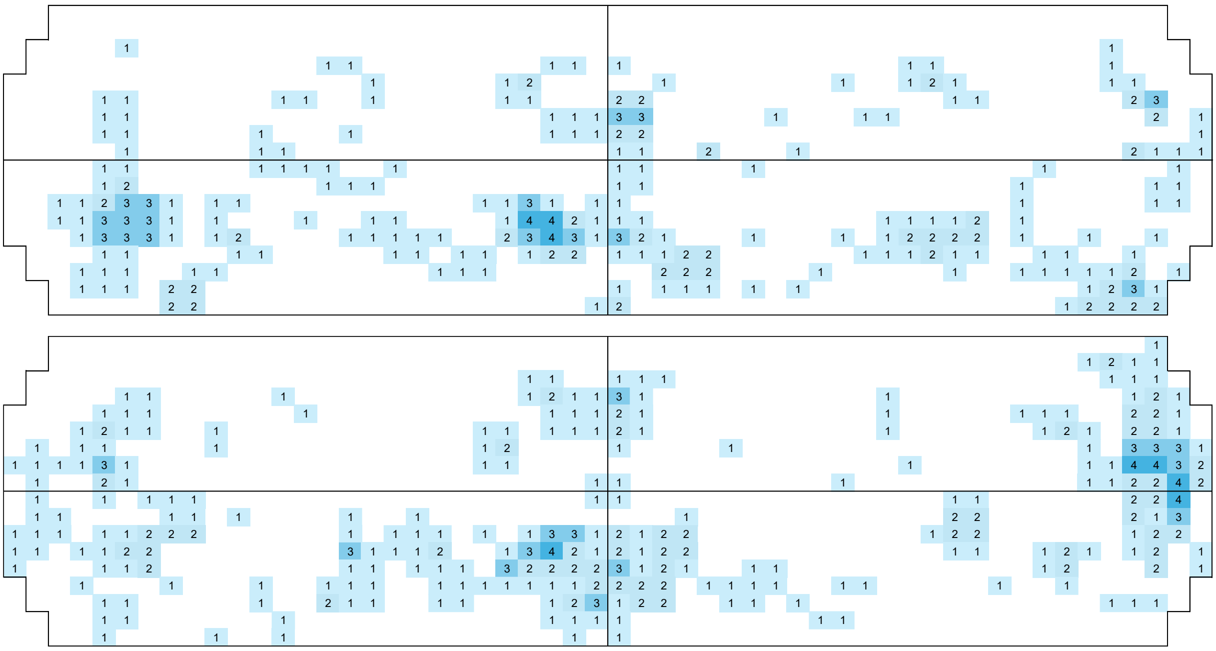


Figure S 24 Hit maps of the h30-d10 Cylinder at a frequency of 15 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

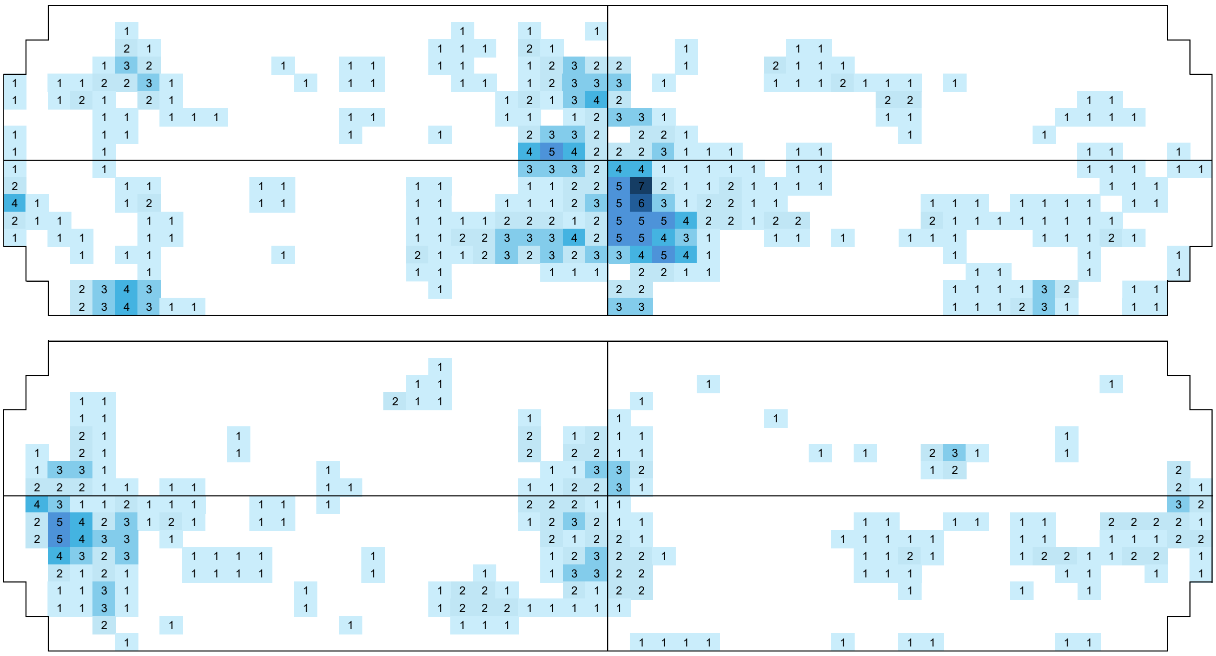


Figure S 25 Hit maps of the h30-d10 Cylinder at a frequency of 20 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

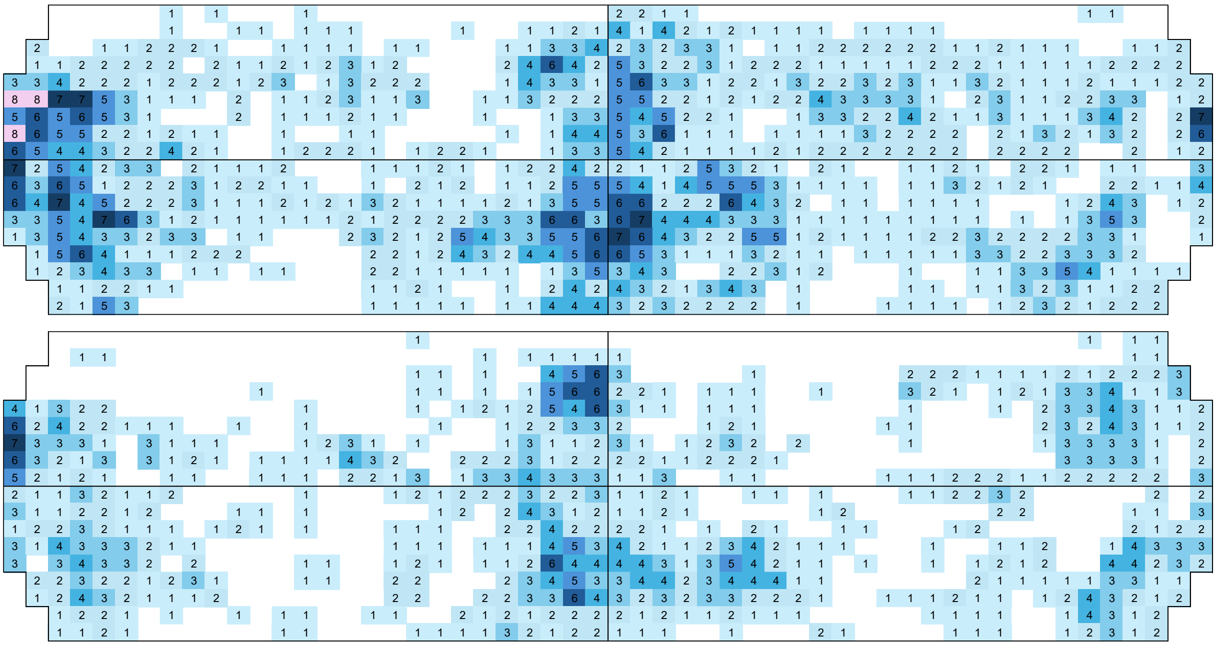


Figure S 26 Hit maps of the h30-d10 Cylinder at a frequency of 30 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

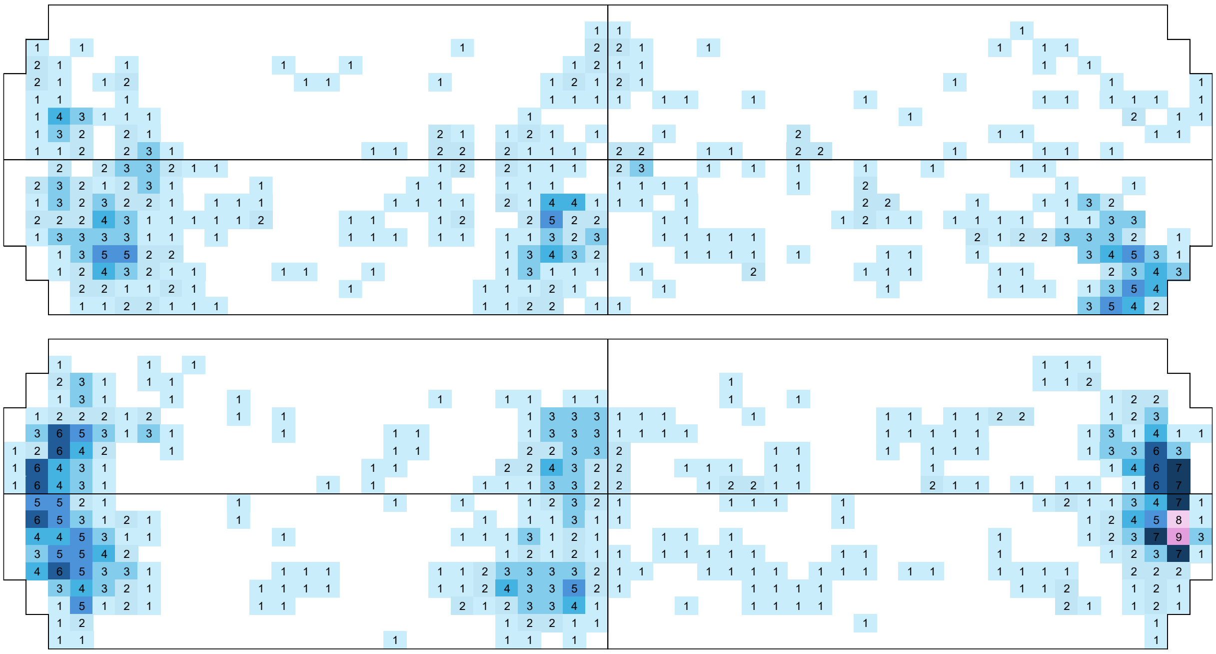


Figure S 27 Hit maps of the h30-d15 Cylinder at a frequency of 15 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

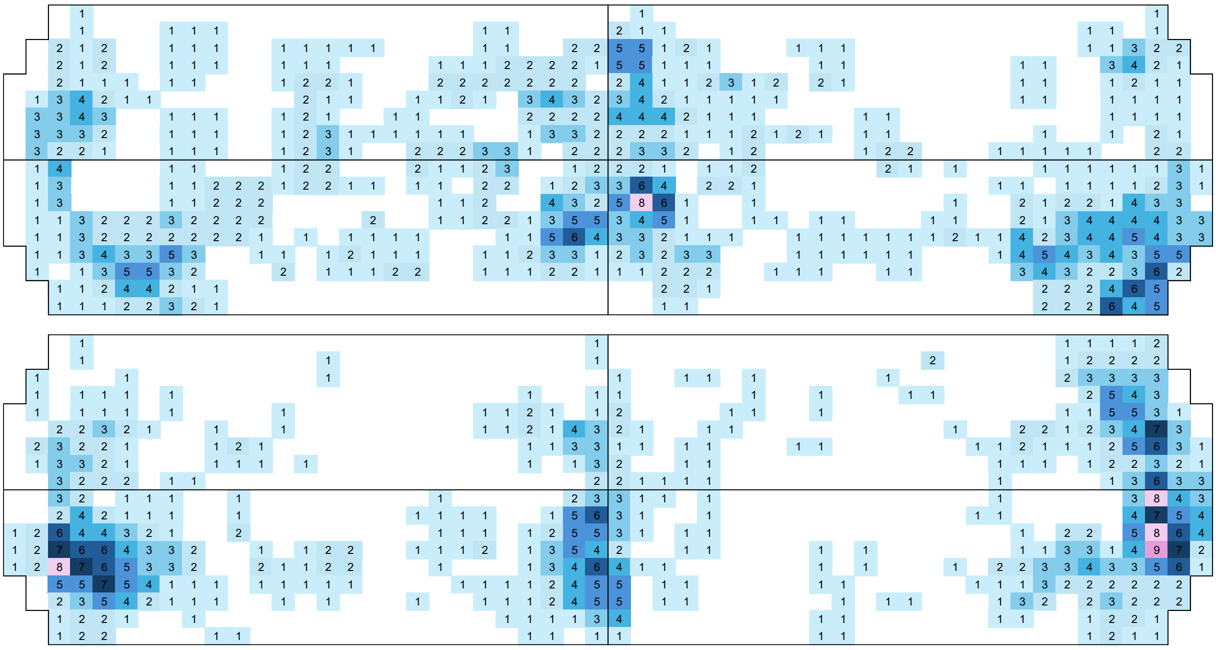


Figure S 28 Hit maps of the h30-d15 Cylinder at a frequency of 20 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

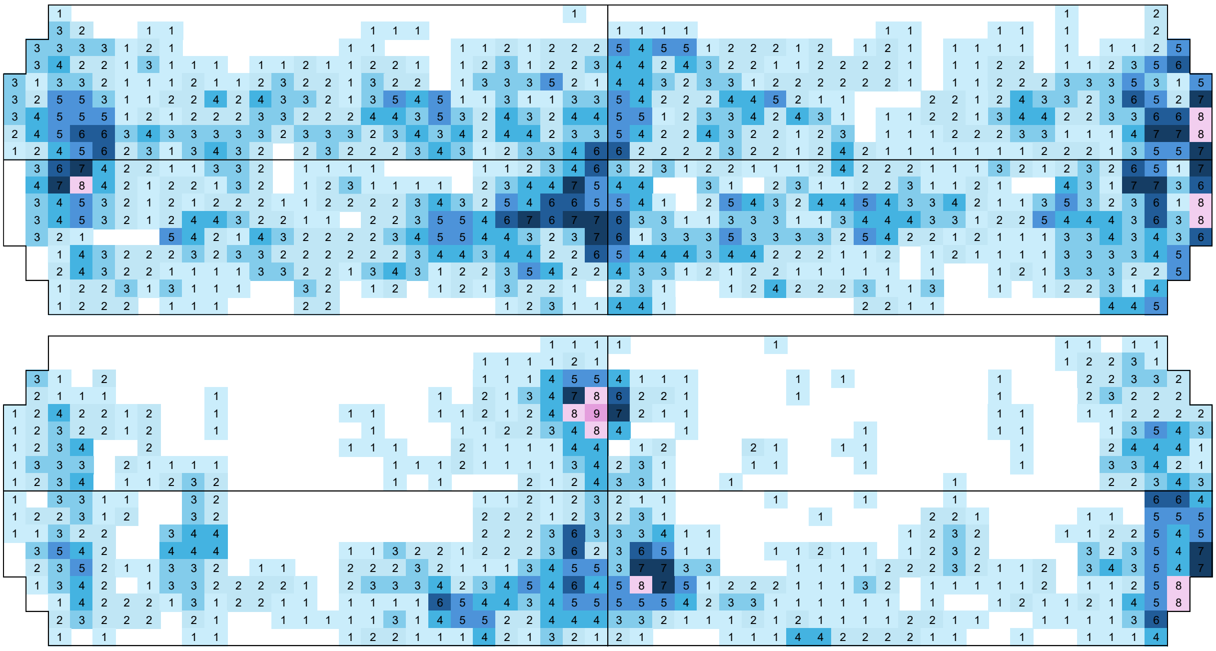


Figure S 29 Hit maps of the h30-d15 Cylinder at a frequency of 30 Hz. Top: Front view; Bottom: Bottom view. The hit counts were prepared by analyzing 6 videos of 0.5 s exposure. All hit numbers correspond to an assigned color.

Table S 4 Hit counts of the h30-d10 and the h30-d15 Cylinders at frequencies of 15, 20, 25, and 30 Hz

	h30-d10			h30-d15		
Frequency	Front View	Bottom View	Overall	Front View	Bottom View	Overall
15	308	364	672	525	678	1203
20	540	351	891	997	833	1830
25	950	662	1612	1436	846	2282
30	1650	1055	2705	2045	1230	3275

6 Time measurements for particle size reduction

As before, the milling times were determined using the Insolido IST636 at 25 Hz. The mill was placed inside of a fully darkened room, and for each measurement 50 mg of copper complex were used. Time measurements were done with the stopwatch function of the Samsung Galaxy S20Fe. The stopwatch was started at the same time as the mixer mill and was stopped at the time when no light reflexes could be observed anymore. For every milling body design the measurement was done twice, and the average of those two measurements was used. All measured and averaged times are shown in Table S5.

Table S 5 Light decay times for the non cylindrical bodies with 50 mg of triboluminescent material at a frequency of 25 Hz

Milling Body	Milling Time 1	Milling Time 2	Average Milling Time [s]
Milling Ball	73.22	75.17	74.195
Cube 5mm[a]	300	300	300
Cube 10mm	161.112	158.29	159.701
Cuboid 15x10mm[a]	300	300	300
Hexagonal Prism h15	81.99	82.1	82.045
Icosahedron	115.74	119.88	117.81
Half Sphere	128.33	132.26	130.295

[a] The experiment was terminated after 300 s.

Cylinders:

Table S 6 Light decay times for the cylindrical bodies with 50 mg of triboluminescent material at a frequency of 25 Hz

	d10			d15		
Cylinder Hight	Milling time 1	Milling Time 2	Average	Milling Time	Milling Time 2	Average
10	75.7	67.18	71.44	44.53	46.56	45.545
15	76.44	74.77	75.605	-	-	
20	59.75	52.06	55.905	28.19	27.12	27.655
25	34.78	35.92	35.35	19	19.62	19.31
30	23.65	26.14	24.895	17.19	16.94	17.065
35	21.43	20.39	20.91	13,6	12.14	12.87
40	27.47	30.11	28.79	16.48	17.89	17.185
45	33.77	38.51	36.14	23.56	22.3	22.93
50	43.93	45.01	44.47	28.03	26.97	27.5

7 Particle size measurements

For measuring of the particle sizes sieves with hole diameters of 3.00 mm, 1.50 mm, 1.00 mm, and 0.75 mm were applied. The measurements were conducted by placing the solids on the sieve and shaking the sieves so that the smaller particles fall through the holes. For the measurement of the starting material, 500 mg of the triboluminescent crystals were put on the biggest sieve, and all sieves were weight after shaking. For the measurements after the particle size degradation, 50 mg of the triboluminescent crystals were loaded to the milling jar, and the mill was used with the milling ball or the h35-d15 cylinder for the times of the light decay measurements (74 s / 13 s). Then, the milled particles were transferred to the 0.75 mm sieve due to already small particles. All measurements are shown below.

Table S 7 Particle size distribution of the triboluminescent crystals before the milling

Particle size	Starting Material		
	empty	full	difference
>3.00 mm	6,3827	6,407	0,0243
1.50 mm-3.00 mm	6,4517	6,554	0,1023
1.00 mm-1.50 mm	6,5214	6,6133	0,0919
0.75 mm-1.00 mm	6,6015	6,7061	0,1046
<0.75 mm	6,7147	6,8733	0,1586

Table S 8 Particle sizes of the triboluminescent crystals after the same milling times as with the light decay measurements for the milling ball and the h35-d15 cylinder

Particle size	Milling Ball 74 s			h35-d15 13 s		
	empty	full	difference	empty	full	difference
0.75 mm-1.00mm	6,1889	6,1944	0,0055	6,3329	6,3335	0,0006
<0.75 mm	6,283	6,2968	0,0138	6,3925	6,4055	0,013

8 Synthesis of 4-bromobenzylthiocyanate (2)[S2]

As test reaction, the substitution of 4-bromobenzylbromide (**1**) with potassium thiocyanate to give 4-bromobenzylthiocyanate (**2**) was selected. The used milling bodies and milling times are given in the main document.

4-Bromobenzylbromide (**1**, 50.0 mg, 0.2 mmol, 1.0 equiv.) and potassium thiocyanate (58.3 mg, 0.6 mmol, 3.0 equiv.) were combined in the 3-D printed milling jar, and the milling body of choice was added. The mixture was shaken at 25 Hz for the given time, between 30 min and 90 min, in the mixer mill IST636 from Insolido. Then, the mixture was dissolved in $CDCl_3$ (1 mL) and filtered over a cotton plug. The conversions were determined by comparing the CH_2 singlet of benzylbromide **1** with the CH_2 singlet of thiocyanate **2**.

NMR

^{1}H-NMR (400 Mhz, $CDCl_3$-*d*): δ = 7.57-7.50 ppm. (m, 2H, $H_{arom.}$), 7.25-7.22 ppm. (m, 2H, $H_{arom.}$), 4.10 ppm. (s, 2H, CH_2)

9 Results of the substitution reaction

In Table S9, the NMR conversions are shown with their corresponding [1]H NMR integrals.

Table S 9 [1]H NMR integrals for the CH2-group signal of 2 with corresponding conversions at a frequency of 25 Hz and reaction times between 30 min and 90 min

	Milling Ball		h35-d10		h35-d15	
	Integral - SCN	Conversion [%]	Integral - SCN	Conversion [%]	Integral - SCN	Conversion [%]
30 min	0,14	12,3	6,74	87,1	9,36	90,4
60 min	0,63	38,7	274,34	99,6	140,35	99,3
90 min	1,96	66,2	77,68	98,8	81,46	98,8

The conversion was determined by setting the NMR integral of the CH_2 group of 4-bromobenzylbromide (**1**) to 1 and analyzing the corresponding CH_2 group of the 4-bromobenzylthiocyanate (**2**). To get the conversion, the following formula was used.

$$Conversion\ \% = \left(1 - \left(\frac{1}{Integral\ \left(CH_{2\ (Thiocyanate)}\right) + 1}\right)\right) \cdot 100\ \%$$

10 References

[S1] F. Marchetti, C. Di Nicola, R. Pettinari, I. Timokhin and C. Pettinari, *J. Chem Educ.,* 2012, **89**, 652-655.

[S2] P. Vogel, S. Figueira, S. Muthukrishnan and J. Mack, *Tetrahedron Lett.,*. 2009, **50**, 55-56.