Electronic Supplementary Material (ESI) for Reaction Chemistry & Engineering.

Supporting Information

Low-Cost, Automated Reaction Screening for Energetic Precursor Cage Compounds by a Benchtop Liquid Handling Robot and Desorption Electrospray Ionization Mass Spectrometry

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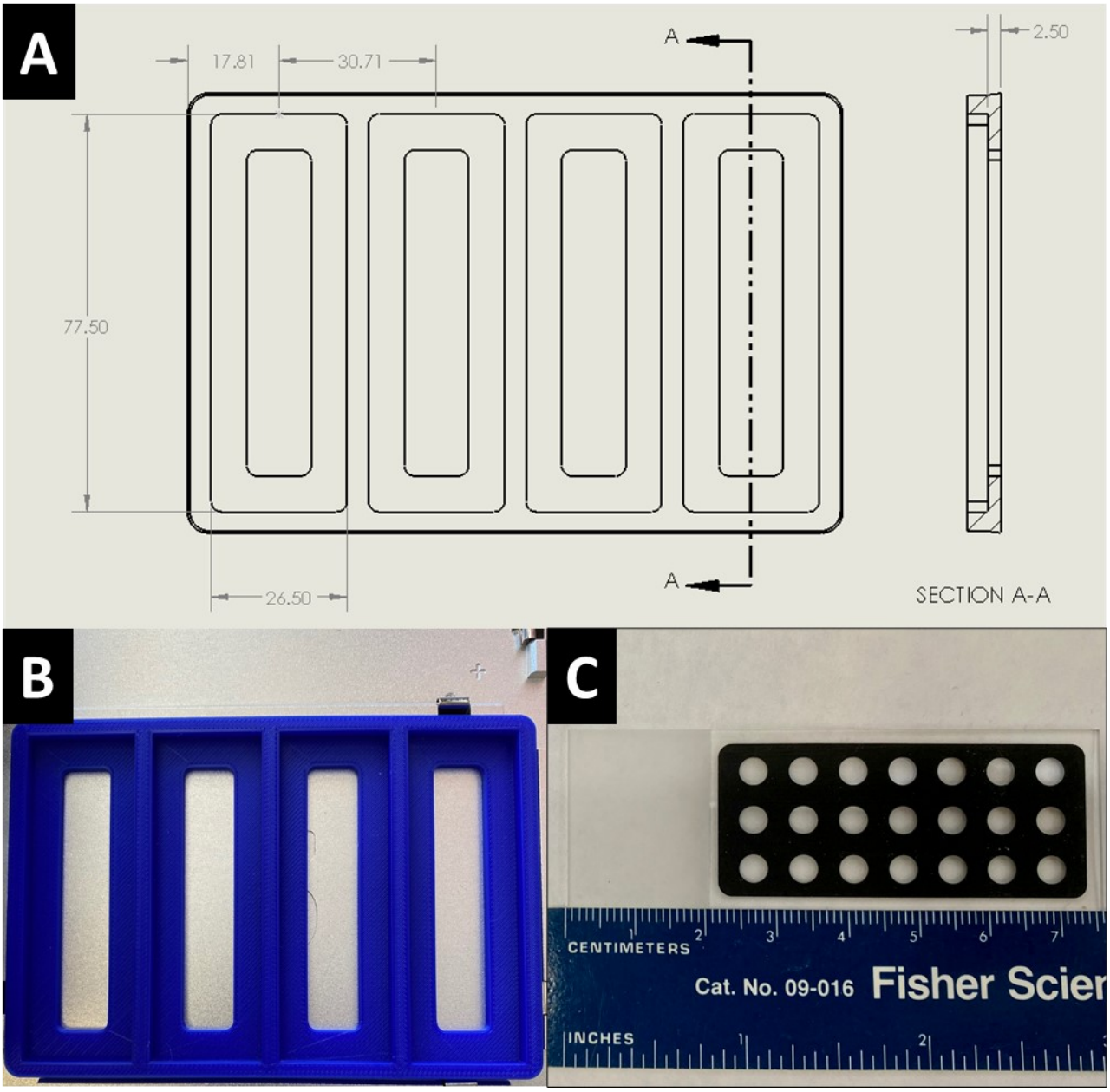

Figure S1. (A) Schematic of the DESI slide holder designed by Opentrons. Dimensions in mm **(B)** Photo of the 3D-printed DESI slide holder. **(C)** Photo of the glass PTFE coated slide.

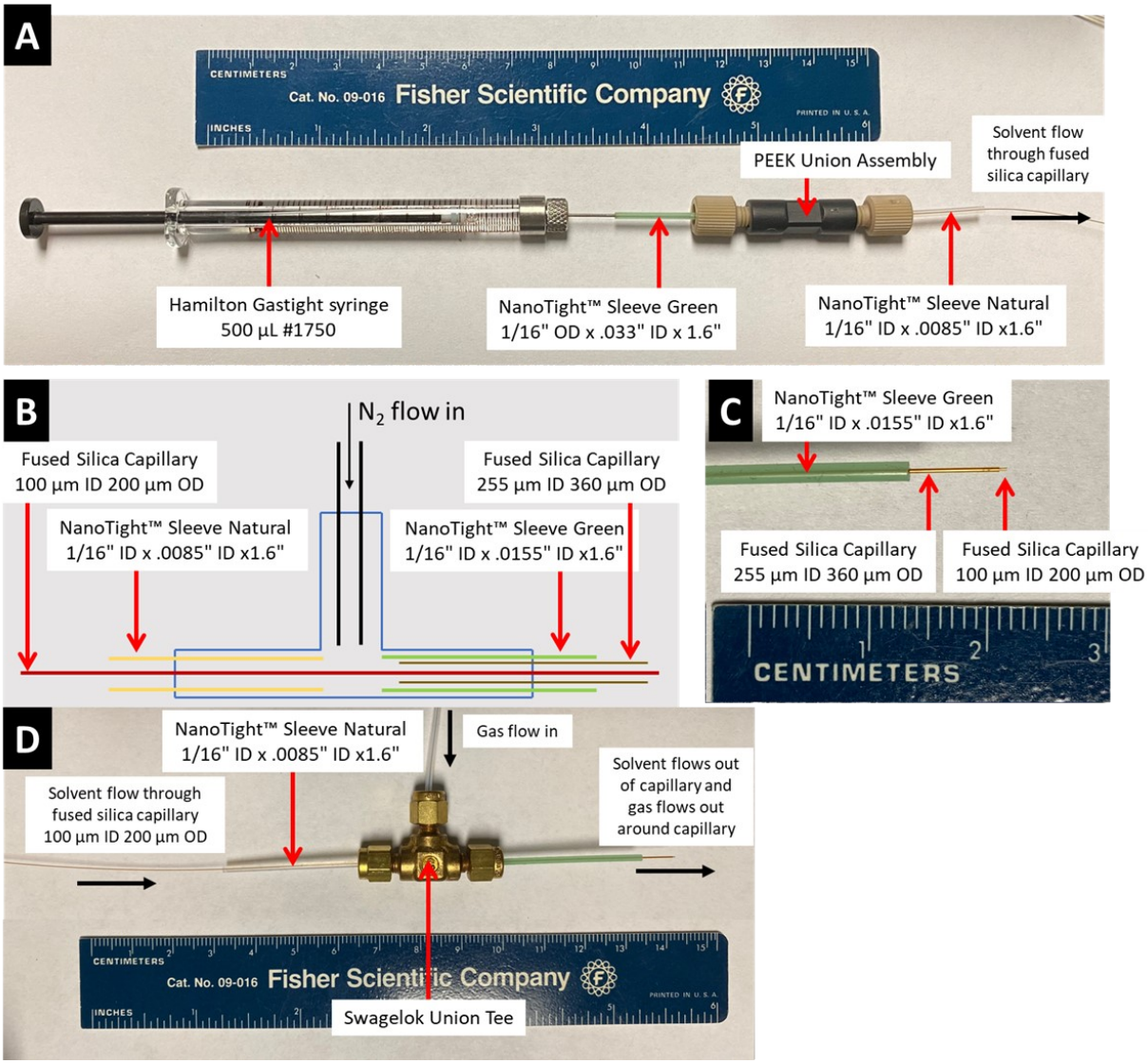

Figure S2. (A) Parts and assembly of the syringe and fittings needed to connect the syringe to the fused silica capillary tubing. **(B)** Schematic drawing of the spray emitter showing how the capillaries are placed within each other and held by the NanoTight™ sleeves. **(C)** Zoomed view of the spray emitter tip to show inner 100x200 µm capillary within 255x360 µm capillary. **(D)** Assembly of parts and fittings needed for the spray emitter.

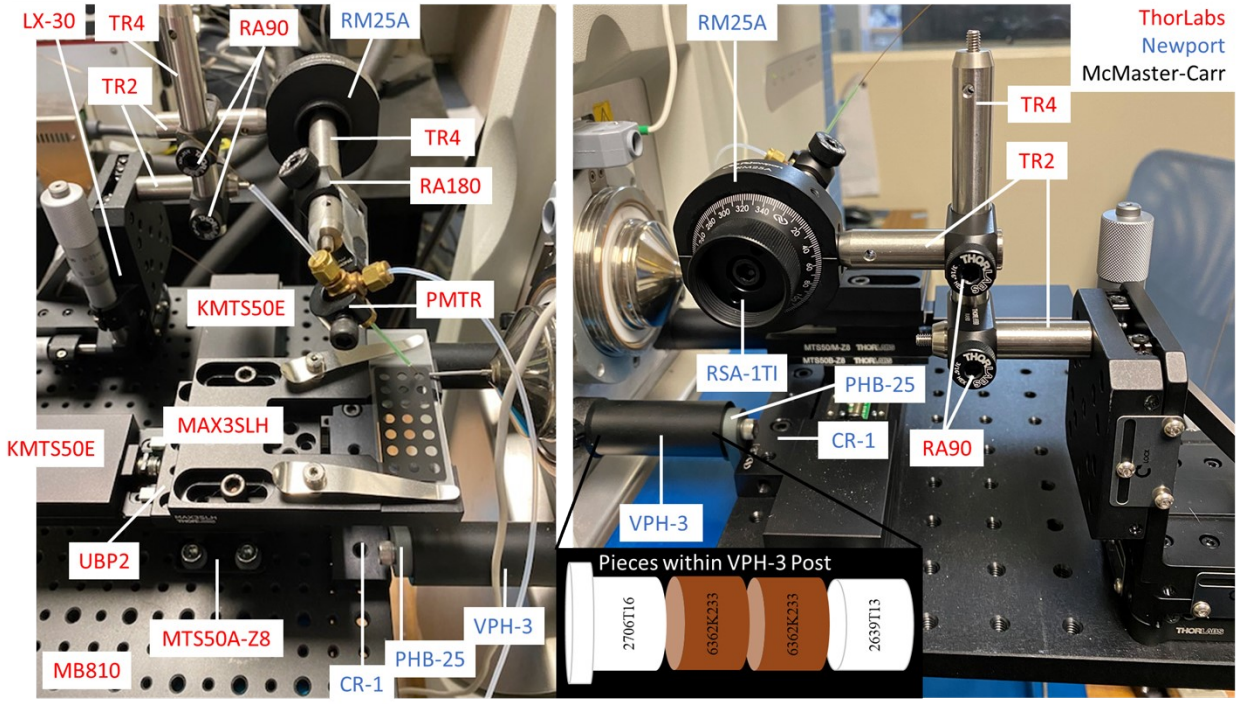

Figure S3. Pictures of the complete DESI stage setup. Part numbers are included for comparison with the full part list and for easy reference when constructing. Red part numbers were ordered from Thorlabs, blue part numbers were ordered from Newport, and black part numbers were ordered from McMaster-Carr.

Table S1. Parts list for the DESI emitter and xy translational stage setup.

	NAME	PART NUMBER	COMPANY	PRICE	QUANTITY		PURPOSE
PIECES NEED TO ATTACH BREADBOARD TO LTQ	PTFE Flanged Sleeve Bearings	2706T16	McMaster-Carr	$6.78	2	$13.56	Fill VPH-3 post holder for tight fit on LTQ pegs
	Rulon LR Sleeve Bearing	6362K233	McMaster-Carr	$5.35	4	$21.40	Fill VPH-3 post holder for tight fit on LTQ pegs
	PTFE Sleeve Bearing	2639T13	McMaster-Carr	$6.43	2	$12.86	Fill VPH-3 post holder for tight fit on LTQ pegs
	Optical Post Holder	VPH-3	Newport	$16.50	2	$33.00	Post holder to attach platform to LTQ
	Post Holder Base	PHB-25	Newport	$5.25	2	$10.50	Connect post holder to construction rail
	Construction Rail	CR-1	Newport	$22.00	1	$22.00	Connect post holder base to breadboard
	Aluminum Breadboard	MSB12	ThorLabs	$227.63	1	$227.63	Breadboard build stage upon
XY STAGE PARTS FOR SLIDE	XY Stage	KMTS50E	ThorLabs	$1,659.87	2	$3,319.74	Motorized translational stage with power supply
	Base Plate for Translation Stages	MTS50A-Z8	ThorLabs	$86.03	1	$86.03	Attach xy stage to breadboard
	Universal Base Plate	UBP2	ThorLabs	$38.69	1	$38.69	Connecting piece between Y stage and slide holder
	Microscopy Slide Holder	MAX3SLH	ThorLabs	$134.73	1	$134.73	Slide holder
PARTS NEEDED TO HOLD DESI EMITTER AND HAVE XYZ/ANGLE CONTROL	Right-Angle End Clamp for Ø1/2" Posts	RA180	ThorLabs	$11.46	1	$11.46	Building blocks for ESI holder
	Right-Angle Clamp for Ø1/2" Posts	RA90	ThorLabs	$10.05	2	$20.10	Building blocks for ESI holder
	Ø1/2" Optical Post, L=2	TR2	ThorLabs	$5.35	2	$10.70	Building blocks for ESI holder
	Ø1/2" Optical Post, L=4	TR4	ThorLabs	$6.05	2	$12.10	Building blocks for ESI holder
	Optic Rotation Mount, 25.4 mm, 2° Graduations	RM25A	Newport	$92.00	1	$92.00	Control angle of DESI sprayer
	Self-Contained XYZ 25 mm Translation Stage, 1/4"-20 Taps	LX-30	ThorLabs	$1,545.00	1	$1,545.00	Control xyz position of DESI sprayer
	Solid Insert, RSP-1T Rotation Stage	RSA-1TI	Newport	$14.00	1	$14.00	Insert for rotation mount to allow post connection
	Component Clamp for Ø1/2" Posts, 1/4"-20 Taps	PMTR	ThorLabs	$26.51	1	$26.51	Connect DESI sprayer to post
ESI EMITTER PARTS	NanoTight™ Sleeve Green 1/16" ID x .0155" ID x 1.6"	F-242	IDEX	$2.66	1	$2.66	Sleeve to hold silica capillary on spray side
	NanoTight™ Sleeve Natural 1/16" ID x .0085" ID x 1.6"	F-239	Cole-Parmer	$2.60	2	$5.20	Sleeve to hold silica capillary from syringe union to spray tee
	NanoTight™ Sleeve Green 1/16" OD x .033" ID x 1.6"	F-247	IDEX	$2.66	1	$2.66	Sleeve to insert syringe
	PEEK ZDV Union Assembly 10-32 Coned	P-704	IDEX	$35.86	1	$35.86	Union to connect syringe and fused silica capillary
	PTFE Tubing	EW-06417-51	Cole-Parmer	$56.00	1	$56.00	Tubing provides N2 gas to emitter from gas tank
	Fused Silica Capillary 100 µm ID x 200 µm OD (L=10m)	1068150021	Molex/Polymicro	$13.26	1	$13.26	Silica tubing for spray solvent
	Fused Silica Capillary 255 µm ID x 360 µm OD (L=10m)	1068150026	Molex/Polymicro	$8.97	1	$8.97	Silica tubing for gas flow around silica tubing used for spray solvent
	Brass Swagelok Tube Fitting, Union Tee, 1/16 in. Tube OD	B-100-3	Swagelok	$44.21	1	$44.21	Union to connect fused silica capillary, gas input, and emitter
	2 cm Extended Capillary (Bent In-House 10°)	SIS20198IS	Scientific Instrument Services	$265.00	1	$265.00	Mass spectrometer inlet
	PTFE Printed Slides, 21 Wells, 4 mm Diameter, 3 in x 1 in (76x25 mm) Box of 100	02289-AB	SPI Supplies	$77.94	1	$77.94	DESI slides for spotting
	High-Purity Line Regulator - Brass	3420 Series	Matheson	$497.48	1	$497.48	Regulator for House N_2 Max 200 PSI

		Total	$6,661.25

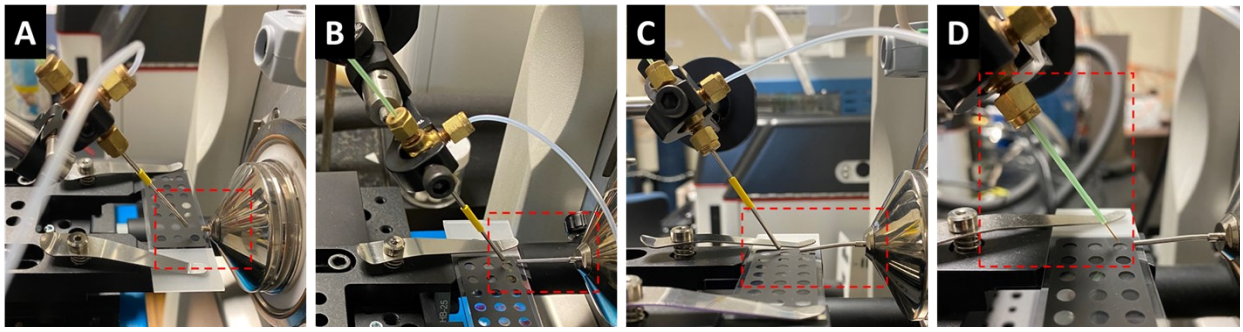

Figure S4. Progression pictures of the emitter and the mass spectrometer inlet. **(A)** The original DESI setup constructed with a stainless-steel tubing emitter, a 100µm ID x 360µm OD capillary and a standard ion transfer capillary. **(B)** The standard ion transfer capillary was replaced with a 2cm extended capillary. **(C)** The extended capillary was bent ~10° towards the slide to improve ion collection. **(D)** The stainless-steel emitter was replaced with a capillary within a capillary setup.

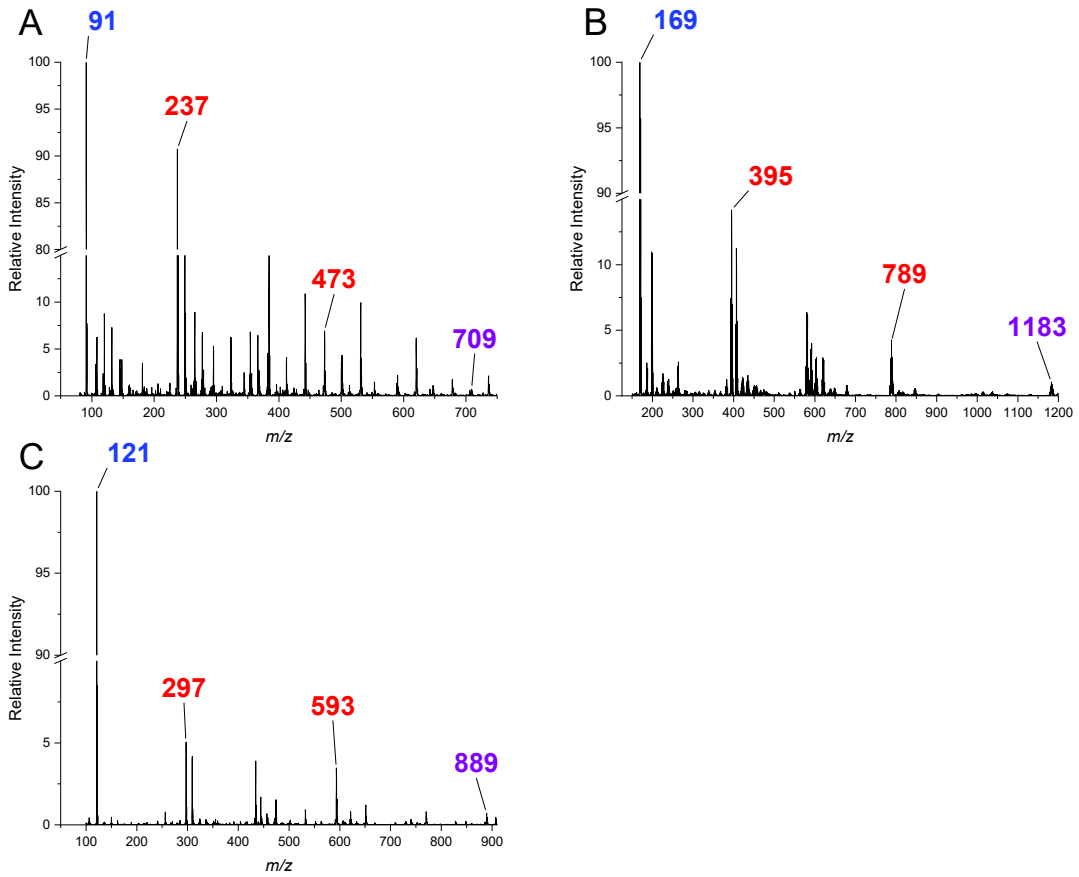

Figure S5. The averaged spectra over one spot on a DESI-MS line scan for screening amine analogs, **(A)** benzylamine, **(B)** bromo-benzylamine, and **(C)** methoxy-benzylamine, for the formation of the energetic precursor cage compounds. Starting materials are indicated with the blue *m/z* labels, intermediates with red *m/z* labels, and products with purple *m/z* labels.

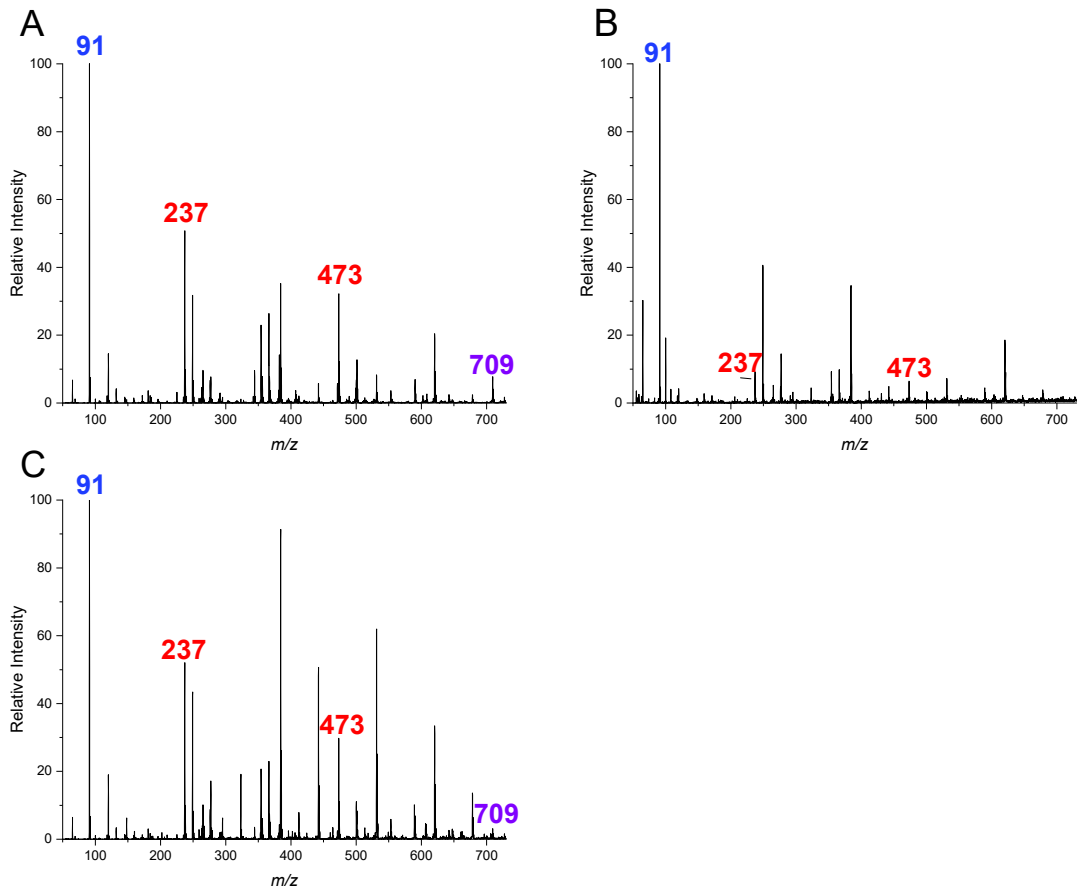

Figure S6. The averaged spectra over one spot on a DESI-MS line scan for screening acid catalysts, **(A)** formic acid, **(B)** trifluoroacetic acid, and **(C)** acetic acid, for the reaction of benzylamine and glyoxal to form the energetic precursor cage compound 2,4,6,8,10,12-hexabenzyl-2,4,6,8,10,12-hexaazaisowurtzitane (HBIW). Starting materials are indicated with the blue *m/z* labels, intermediates with red *m/z* labels, and products with purple *m/z* labels.