

Conversion of Primary and Secondary Iodo- and Bromopropanes, Pt. 1

Press Text: 4249p11

Reference: NB.4172p99-100, NB.4266p5

1-Iodopropane – These experiments qualitatively demonstrate that after 20 minutes in the 110°C headspace oven 1-iodopropane was fully converted to the chloro- product. 1mM HCl in DMAc was the diluent. It is expected that this would be observed for all primary alkyl iodides.

2-Iodopropane – 2-iodopropane demonstrated nearly complete conversion, however some (~2% by peak area) 2-iodopropane was still observed. No evidence of Sn1 substitution was observed where endogenous water could allow formation of 2-propanol (IPA). A follow-up experiment should study increasing both the time in the headspace oven and the concentration of HCl in the diluent (see [12]). It is plausible that steric hinderance reduces the likelihood of chloride favorably colliding wit the opposite side of the iodo-carbon, leading to a slower conversion rate.

1-Bromopropane – 1-bromopropane showed ~6% (by peak area) remaining indicating that this technique is feasible for primary alkyl bromides as well. As with 2-iodopropane, the time in the headspace oven and concentration of HCl should be studied to see if complete conversion can be achieved in a timeframe that remains practical for routine analysis (see [12]). Compared to the 1-iodopropane conversion rate, bromide is a poorer leaving group suggesting that while steric hinderance is not an issue, more direct collisions are seemingly required to achieve the activation energy needed to induce the Sn2 reaction.

2-Bromopropane – Only 30% (by peak area) of the 2-bromopropane was observed to convert to 2-chloropropane. No evidence of Sn1 substitution is observed either. These results would seem to suggest that the 2-bromopropane is relatively stable, potentially enough so to not warrant the need for derivation in the first place. However, the concentration of HCL may still allow the reaction to be driven to completion.

Sequence Overview

Sequence Details

| | | | |
|---------------------------|---------------------------------|--------------------|---------------------------|
| Name: | 230504bz | Created On: | 04/May/23 09:44:25 |
| Directory: | E511\Finkelstein\R&D | Created By: | bzielke |
| Data Vault: | chrom72 | Updated On: | 21/Sep/23 09:52:14 |
| No. of Injections: | 12 | Updated By: | bzielke |

Injection Details

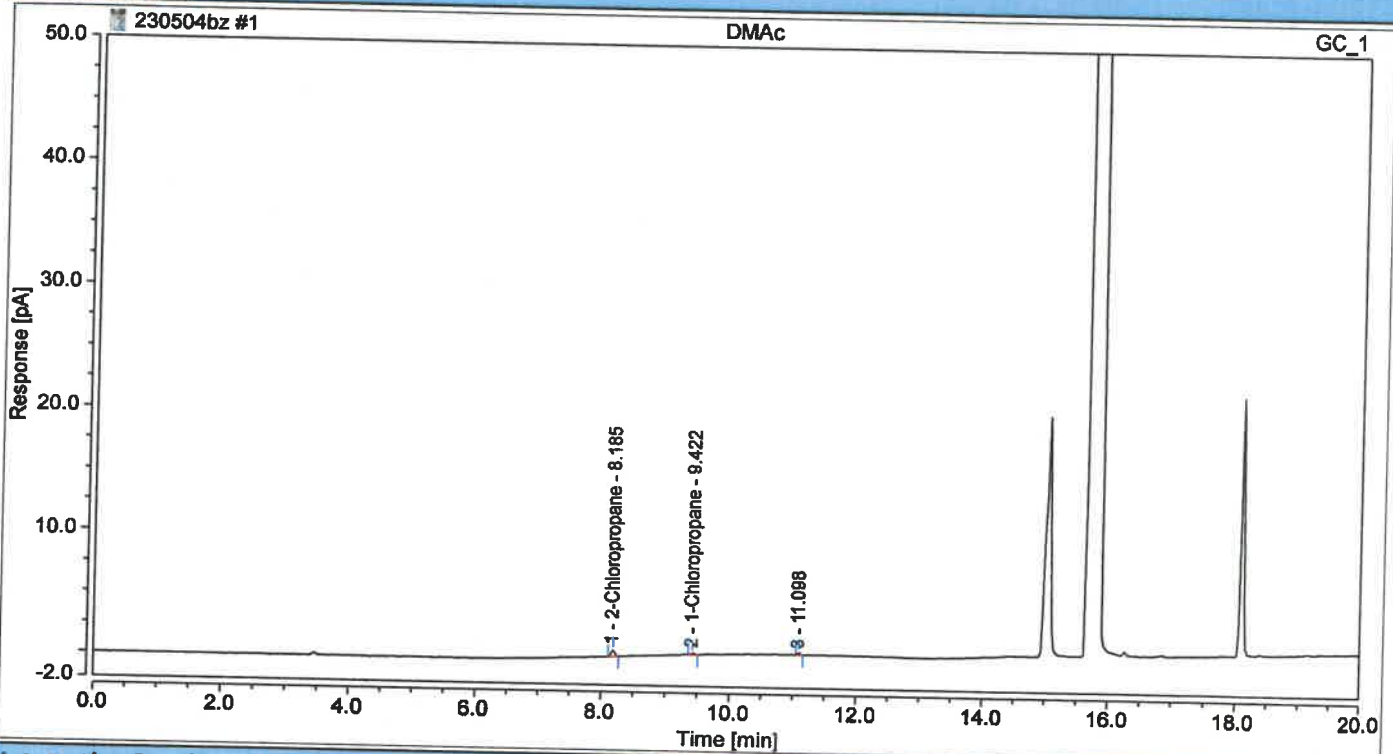
| No. | Injection Name | Position | Type | Proc. Method Version | Inject Time | Status |
|-----|----------------------|----------|---------|----------------------|--------------------|----------|
| 1 | DMAc | 1 | Unknown | 3 | 04/May/23 14:12:01 | Finished |
| 2 | DMAc | 2 | Unknown | 3 | 04/May/23 14:42:05 | Finished |
| 3 | 1-Chloropropane ID | 3 | Unknown | 3 | 04/May/23 15:12:05 | Finished |
| 4 | 2-Chloropropane ID | 4 | Unknown | 3 | 04/May/23 15:42:05 | Finished |
| 5 | 1-Iodopropane ID | 5 | Unknown | 3 | 04/May/23 16:12:05 | Finished |
| 6 | 2-Iodopropane ID | 6 | Unknown | 3 | 04/May/23 16:42:05 | Finished |
| 7 | 1-Bromopropane ID | 7 | Unknown | 3 | 04/May/23 17:12:06 | Finished |
| 8 | 2-Bromopropane ID | 8 | Unknown | 3 | 04/May/23 17:42:06 | Finished |
| 9 | 1-Iodopropane w/HCl | 9 | Unknown | 3 | 04/May/23 18:12:06 | Finished |
| 10 | 2-Iodopropane w/HCl | 10 | Unknown | 3 | 04/May/23 18:42:06 | Finished |
| 11 | 1-Bromopropane w/HCl | 11 | Unknown | 3 | 04/May/23 19:12:05 | Finished |
| 12 | 2-Bromopropane w/HCl | 12 | Unknown | 3 | 04/May/23 19:42:05 | Finished |

Chromatogram and Results

Injection Details

| | | | |
|-----------------------------|-----------------|--------------------------|--------|
| Injection Name: | DMAc | Run Time (min): | 20.00 |
| Vial Number: | 1 | Injection Volume: | 1.00 |
| Injection Type: | Unknown | Channel: | GC_1 |
| Comment: | | Wavelength: | n.a. |
| Instrument Method: | Finkelstein 1 | Bandwidth: | n.a. |
| Processing Method: | Quantitative | Dilution Factor: | 1.0000 |
| Injection Date/Time: | 04/May/23 14:12 | Sample Weight: | 1.0000 |

Chromatogram



Integration Results

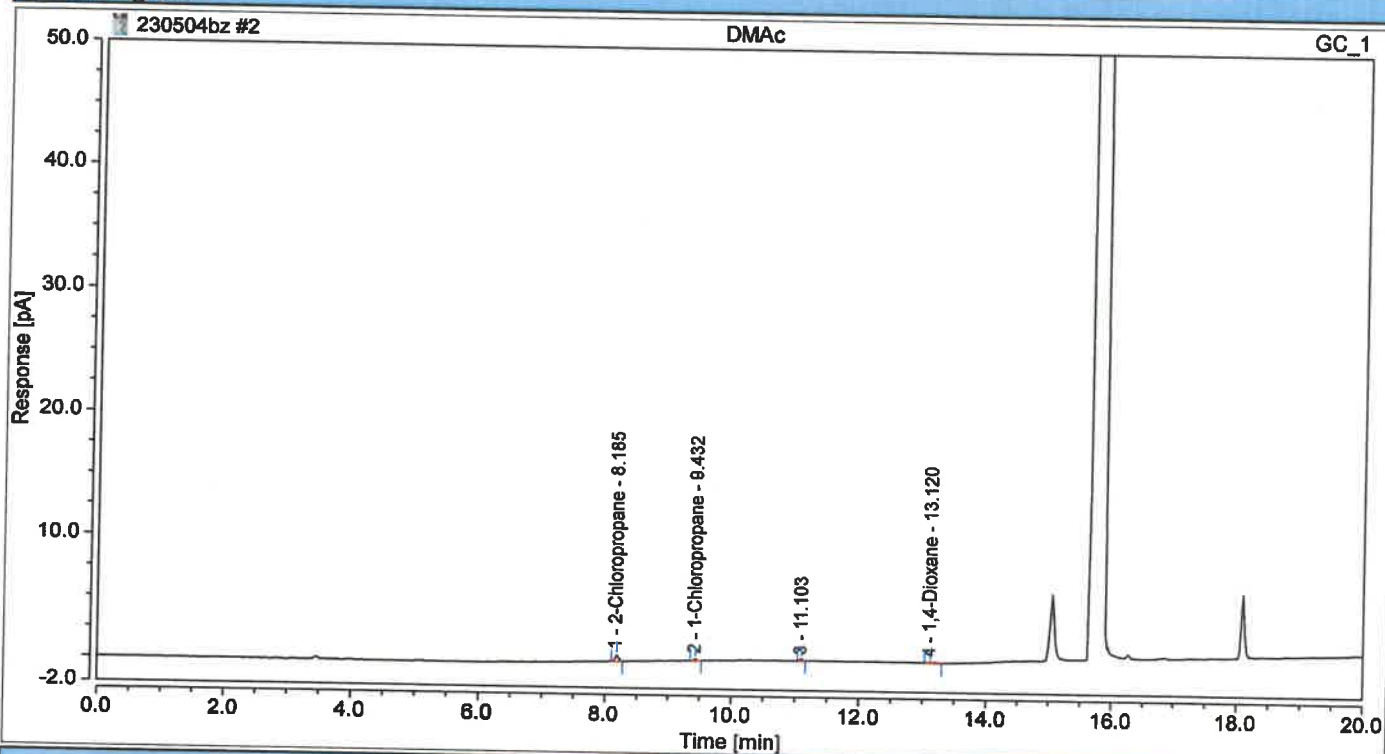
| No. | Peak Name | Retention Time min | Area pA*min | Height pA | Resolution (USP) To Next | Tailing USP | S/N |
|--------|-----------------|-----------------------|----------------|--------------|-----------------------------|-------------|------|
| 1 | 2-Chloropropane | 8.185 | 0.03477 | 0.552 | 14.3 | 0.9 | 40 |
| 2 | 1-Chloropropane | 9.422 | 0.00772 | 0.130 | 22.3 | 1.0 | 9 |
| n.a. | 2-Bromopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 3 | | 11.098 | 0.00912 | 0.183 | n.a. | 1.0 | 11 |
| n.a. | 1-Bromopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| n.a. | 2-Iodopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| n.a. | 1,4-Dioxane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| n.a. | 1-Iodopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| n.a. | DMAc | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| Total: | | | 0.052 | 0.865 | 36.67 | 2.87 | |

Chromatogram and Results

Injection Details

| | | | |
|----------------------|-----------------|-------------------|--------|
| Injection Name: | DMAc | Run Time (min): | 20.00 |
| Vial Number: | 2 | Injection Volume: | 1.00 |
| Injection Type: | Unknown | Channel: | GC_1 |
| Comment: | | Wavelength: | n.a. |
| Instrument Method: | Finkelstein 1 | Bandwidth: | n.a. |
| Processing Method: | Quantitative | Dilution Factor: | 1.0000 |
| Injection Date/Time: | 04/May/23 14:42 | Sample Weight: | 1.0000 |

Chromatogram



Integration Results

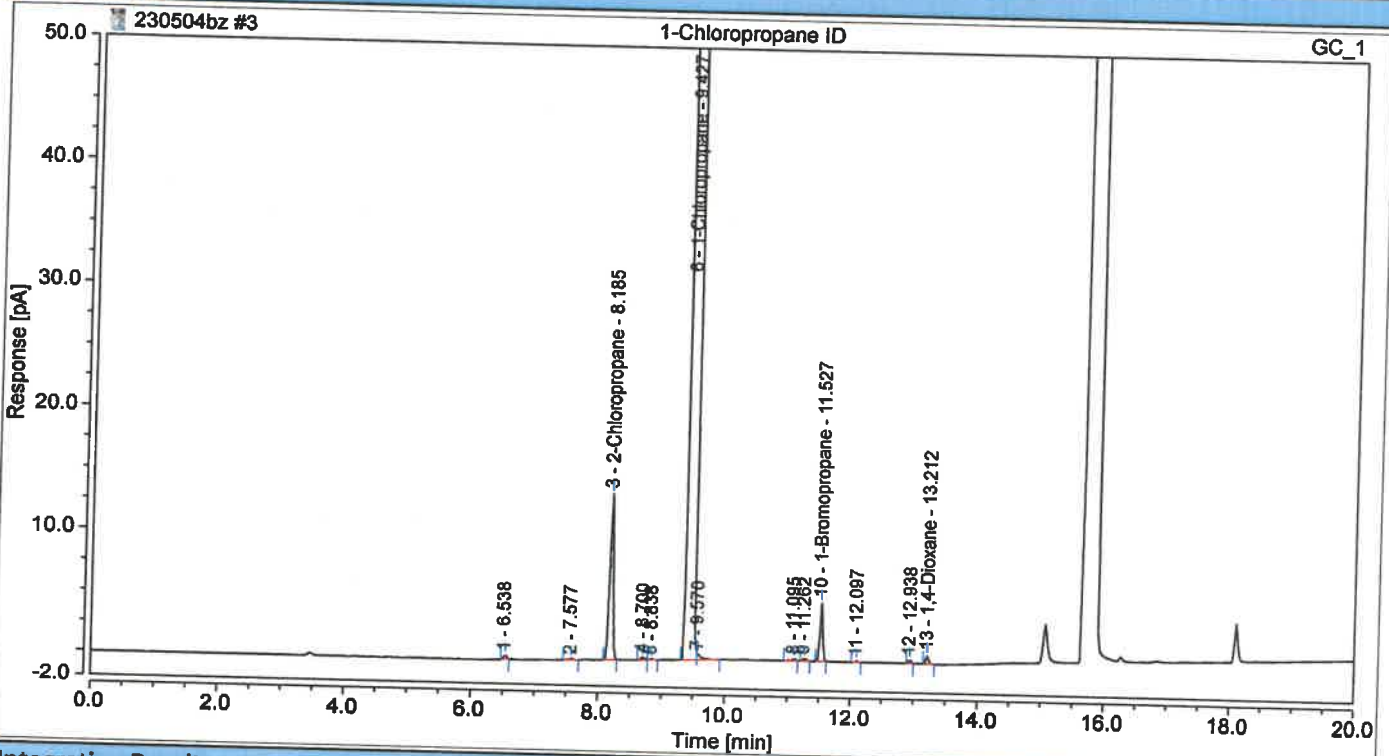
| No. | Peak Name | Retention Time min | Area pA*min | Height pA | Resolution (USP) To Next | Tailing USP | S/N |
|--------|-----------------|-----------------------|----------------|--------------|-----------------------------|-------------|------|
| 1 | 2-Chloropropane | 8.185 | 0.03433 | 0.540 | 13.9 | 1.0 | 25 |
| 2 | 1-Chloropropane | 9.432 | 0.00771 | 0.129 | 19.3 | 1.1 | 10 |
| n.a. | 2-Bromopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 3 | 1-Bromopropane | 11.103 | 0.00907 | 0.175 | 25.4 | 0.9 | 14 |
| n.a. | 2-Iodopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| n.a. | 1-Iodopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 4 | 1,4-Dioxane | 13.120 | 0.00905 | 0.065 | n.a. | 1.5 | 4 |
| n.a. | 1-Iodopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| n.a. | DMAc | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| Total: | | | 0.060 | 0.909 | 58.64 | 4.52 | |

Chromatogram and Results

Injection Details

| | | | |
|----------------------|--------------------|-------------------|--------|
| Injection Name: | 1-Chloropropane ID | Run Time (min): | 20.00 |
| Vial Number: | 3 | Injection Volume: | 1.00 |
| Injection Type: | Unknown | Channel: | GC_1 |
| Comment: | | Wavelength: | n.a. |
| Instrument Method: | Finkelstein 1 | Bandwidth: | n.a. |
| Processing Method: | Quantitative | Dilution Factor: | 1.0000 |
| Injection Date/Time: | 04/May/23 15:12 | Sample Weight: | 1.0000 |

Chromatogram



Integration Results

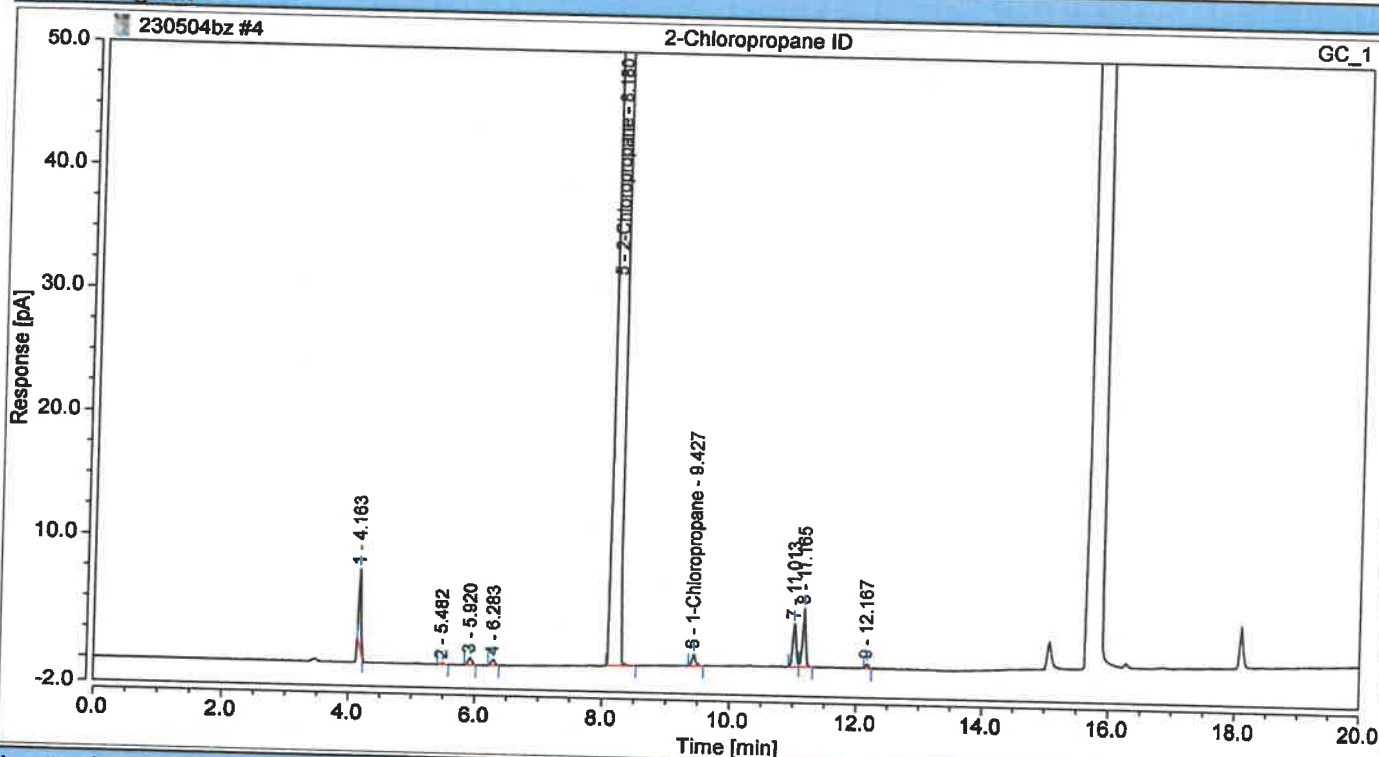
| No. | Peak Name | Retention Time min | Area pA*min | Height pA | Resolution (USP) To Next | Tailing USP | S/N |
|--------|-----------------|-----------------------|----------------|--------------|-----------------------------|-------------|--------|
| 1 | | 6.538 | 0.01992 | 0.349 | 11.5 | 0.9 | 17 |
| 2 | | 7.577 | 0.00958 | 0.114 | 6.2 | 0.9 | 4 |
| 3 | 2-Chloropropane | 8.185 | 0.85594 | 13.518 | 5.1 | 1.0 | 720 |
| 4 | | 8.700 | 0.01590 | 0.247 | 1.4 | n.a. | 20 |
| 5 | | 8.838 | 0.00563 | 0.084 | 6.6 | n.a. | 7 |
| 6 | 1-Chloropropane | 9.427 | 261.12627 | 4683.282 | 2.4 | 1.1 | 361786 |
| 7 | | 9.570 | 0.04119 | 0.471 | 26.3 | n.a. | n.a. |
| n.a. | 2-Bromopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 8 | | 11.095 | 0.01358 | 0.184 | 1.8 | 0.7 | 10 |
| 9 | | 11.262 | 0.01449 | 0.196 | 2.8 | 1.2 | 11 |
| 10 | 1-Bromopropane | 11.527 | 0.23965 | 4.744 | 7.4 | 1.0 | 410 |
| 11 | | 12.097 | 0.00716 | 0.119 | 11.5 | 0.9 | 11 |
| n.a. | 2-Iodopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 12 | | 12.938 | 0.01333 | 0.288 | 3.7 | 0.9 | 29 |
| 13 | 1,4-Dioxane | 13.212 | 0.03364 | 0.682 | n.a. | 1.1 | 52 |
| n.a. | 1-Iodopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| n.a. | DMAC | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| Total: | | | 262.396 | 4704.278 | 86.61 | 9.79 | |

Chromatogram and Results

Injection Details

| | | | |
|-----------------------------|--------------------|--------------------------|--------|
| Injection Name: | 2-Chloropropane ID | Run Time (min): | 20.00 |
| Vial Number: | 4 | Injection Volume: | 1.00 |
| Injection Type: | Unknown | Channel: | GC_1 |
| Comment: | | Wavelength: | n.a. |
| Instrument Method: | Finkelstein 1 | Bandwidth: | n.a. |
| Processing Method: | Quantitative | Dilution Factor: | 1.0000 |
| Injection Date/Time: | 04/May/23 15:42 | Sample Weight: | 1.0000 |

Chromatogram



Integration Results

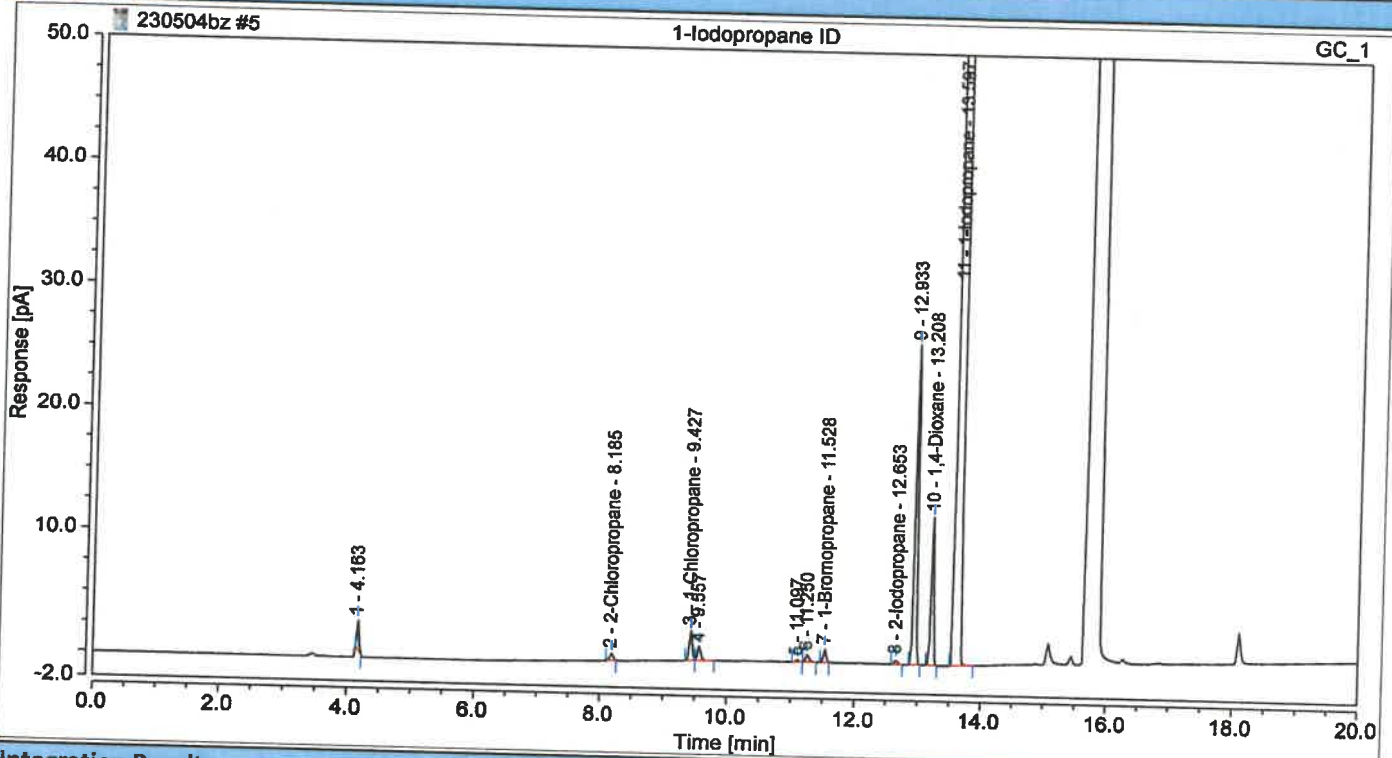
| No. | Peak Name | Retention Time min | Area pA*min | Height pA | Resolution (USP) To Next | Tailing USP | S/N |
|---------------|-----------------|-----------------------|----------------|-----------------|-----------------------------|-------------|--------|
| 1 | | 4.163 | 0.27328 | 6.297 | 18.2 | 1.1 | 86 |
| 2 | | 5.482 | 0.00609 | 0.086 | 5.0 | 0.9 | 10 |
| 3 | | 5.920 | 0.04141 | 0.613 | 3.6 | 1.0 | 79 |
| 4 | | 6.283 | 0.03203 | 0.494 | 19.0 | 1.0 | 46 |
| 5 | 2-Chloropropane | 8.160 | 212.57054 | 3323.153 | 13.1 | 1.1 | 164459 |
| 6 | 1-Chloropropane | 9.427 | 0.05849 | 0.942 | 17.3 | 1.4 | 79 |
| n.a. | 2-Bromopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 7 | | 11.013 | 0.20884 | 3.560 | 1.7 | n.a. | 211 |
| 8 | | 11.165 | 0.27141 | 4.764 | 12.1 | 0.9 | 337 |
| n.a. | 1-Bromopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 9 | | 12.167 | 0.02027 | 0.414 | n.a. | 1.1 | 25 |
| n.a. | 2-Iodopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| n.a. | 1,4-Dioxane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| n.a. | 1-Iodopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| n.a. | DMAc | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| Total: | | | 213.482 | 3340.323 | 89.98 | 8.63 | |

Chromatogram and Results

Injection Details

| | | | |
|-----------------------------|-------------------------|--------------------------|---------------|
| Injection Name: | 1-Iodopropane ID | Run Time (min): | 20.00 |
| Vial Number: | 5 | Injection Volume: | 1.00 |
| Injection Type: | Unknown | Channel: | GC_1 |
| Comment: | | Wavelength: | n.a. |
| Instrument Method: | Finkelstein 1 | Bandwidth: | n.a. |
| Processing Method: | Quantitative | Dilution Factor: | 1.0000 |
| Injection Date/Time: | 04/May/23 16:12 | Sample Weight: | 1.0000 |

Chromatogram



Integration Results

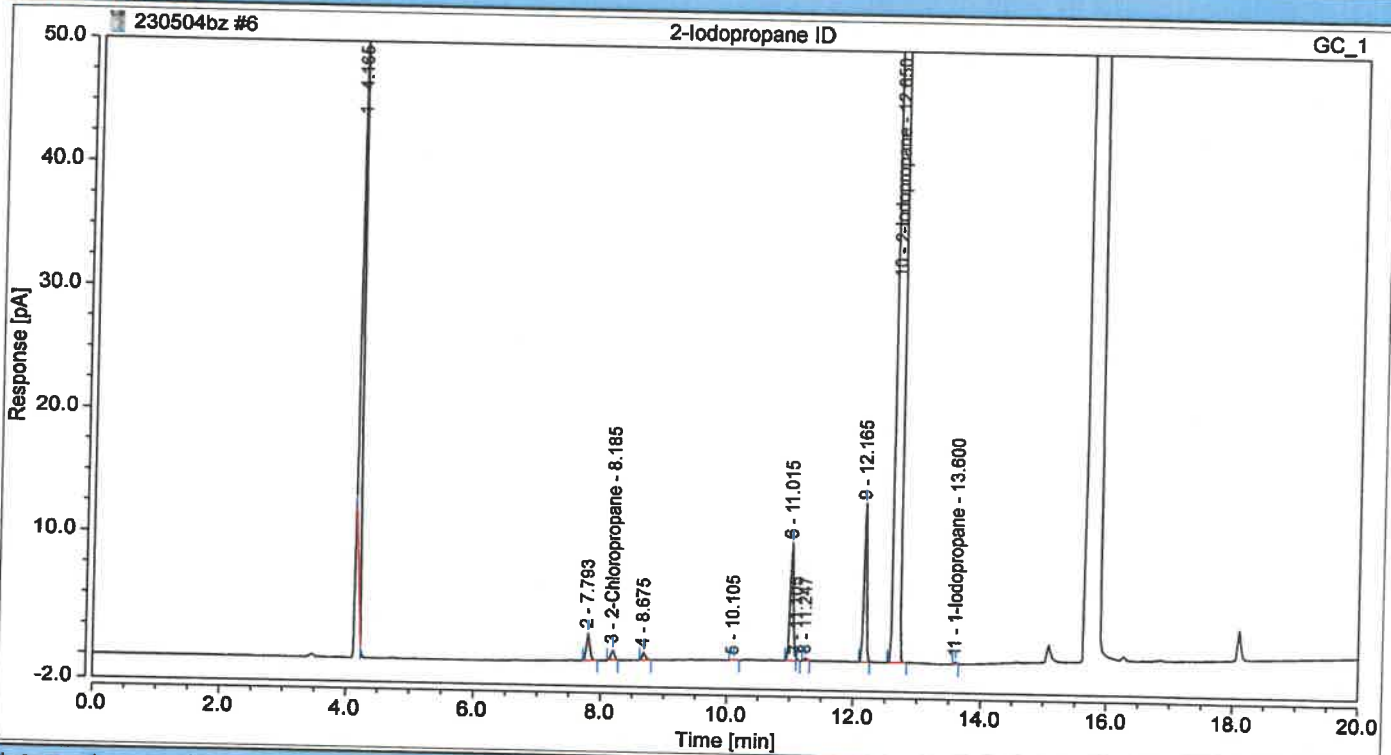
| No. | Peak Name | Retention Time min | Area pA*min | Height pA | Resolution (USP) To Next | Tailing USP | S/N |
|--------|-----------------|-----------------------|----------------|--------------|-----------------------------|-------------|-------|
| 1 | | 4.163 | 0.10871 | 2.500 | 47.4 | 1.1 | 68 |
| 2 | 2-Chloropropane | 8.185 | 0.03918 | 0.621 | 13.2 | 1.0 | 41 |
| 3 | 1-Chloropropane | 9.427 | 0.13709 | 2.451 | 1.4 | 1.0 | 191 |
| 4 | | 9.557 | 0.07540 | 1.209 | 16.6 | 1.3 | 88 |
| n.a. | 2-Bromopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 5 | | 11.097 | 0.01052 | 0.190 | 1.7 | 0.9 | 17 |
| 6 | | 11.250 | 0.03842 | 0.627 | 3.2 | 1.5 | 57 |
| 7 | 1-Bromopropane | 11.528 | 0.05628 | 1.096 | 13.3 | 1.0 | 86 |
| 8 | 2-Iodopropane | 12.653 | 0.02137 | 0.390 | 3.5 | 1.1 | 44 |
| 9 | | 12.933 | 1.22708 | 26.011 | 3.7 | 1.0 | 3771 |
| 10 | 1,4-Dioxane | 13.208 | 0.54866 | 12.000 | 5.1 | 1.0 | 898 |
| 11 | 1-Iodopropane | 13.597 | 61.79004 | 1256.871 | n.a. | 1.1 | 94018 |
| n.a. | DMAC | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| Total: | | | 64.053 | 1303.967 | 109.16 | 12.03 | |

Chromatogram and Results

Injection Details

| | | | |
|----------------------|------------------|-------------------|--------|
| Injection Name: | 2-Iodopropane ID | Run Time (min): | 20.00 |
| Vial Number: | 6 | Injection Volume: | 1.00 |
| Injection Type: | Unknown | Channel: | GC_1 |
| Comment: | | Wavelength: | n.a. |
| Instrument Method: | Finkelstein 1 | Bandwidth: | n.a. |
| Processing Method: | Quantitative | Dilution Factor: | 1.0000 |
| Injection Date/Time: | 04/May/23 16:42 | Sample Weight: | 1.0000 |

Chromatogram



Integration Results

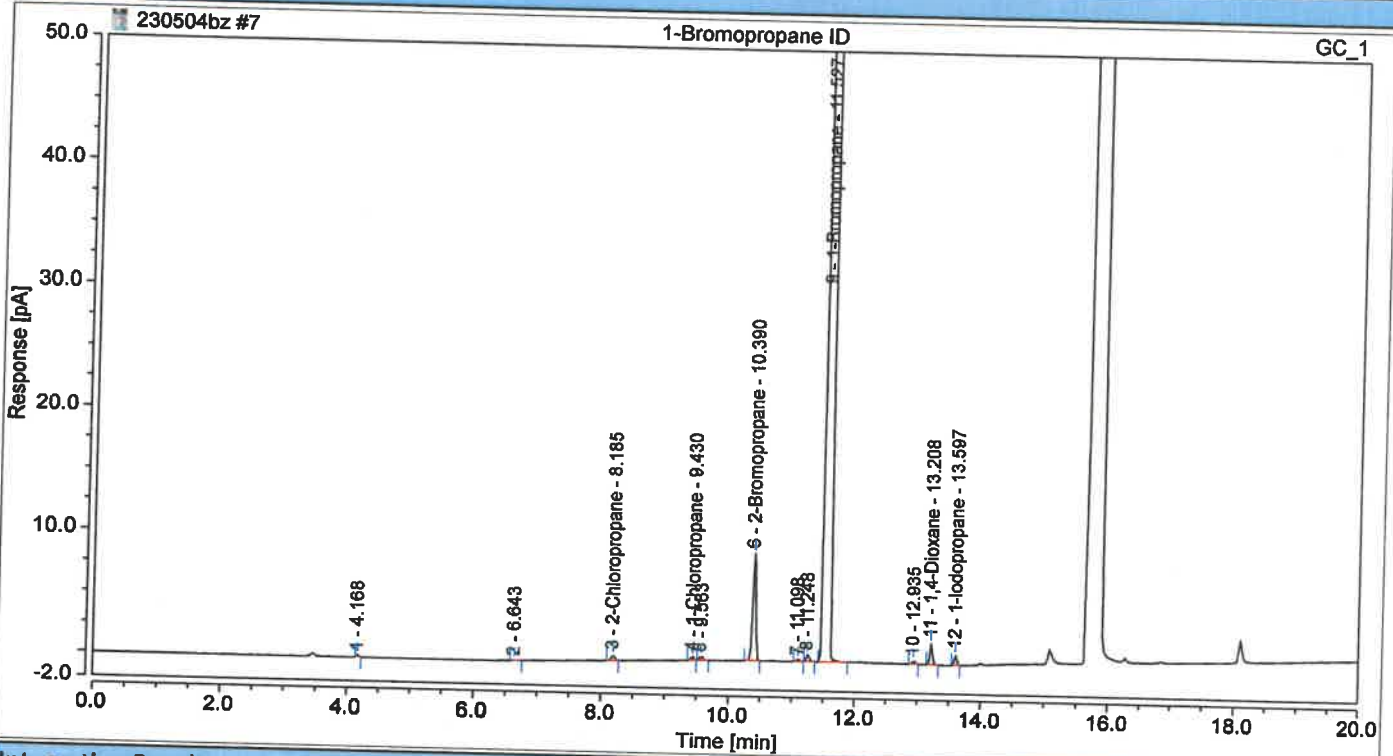
| No. | Peak Name | Retention Time min | Area pA*min | Height pA | Resolution (USP) To Next | Tailing USP | S/N |
|--------|-----------------|-----------------------|----------------|--------------|-----------------------------|-------------|-------|
| 1 | | 4.165 | 2.04177 | 46.280 | 42.6 | 1.1 | 115 |
| 2 | | 7.793 | 0.14139 | 2.220 | 3.9 | 1.1 | 81 |
| 3 | 2-Chloropropane | 8.185 | 0.05334 | 0.841 | 5.2 | 1.0 | 82 |
| 4 | | 8.675 | 0.03823 | 0.650 | 17.9 | 1.3 | 55 |
| n.a. | 1-Chloropropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 5 | | 10.105 | 0.00435 | 0.069 | 11.1 | 1.1 | 2 |
| n.a. | 2-Bromopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 6 | | 11.015 | 0.56269 | 9.609 | 1.9 | 1.0 | 630 |
| 7 | | 11.105 | 0.00498 | 0.190 | 3.3 | n.a. | n.a. |
| 8 | | 11.247 | 0.01120 | 0.226 | 11.5 | 1.0 | 16 |
| n.a. | 1-Bromopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 9 | | 12.165 | 0.64230 | 12.905 | 6.0 | 1.0 | 1044 |
| 10 | 2-Iodopropane | 12.650 | 60.94402 | 1168.938 | 12.7 | 1.1 | 90367 |
| n.a. | 1,4-Dioxane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 11 | 1-Iodopropane | 13.600 | 0.00712 | 0.151 | n.a. | 0.9 | 14 |
| n.a. | DMAC | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| Total: | | | 64.451 | 1242.079 | 116.12 | 10.65 | |

Chromatogram and Results

Injection Details

| | | | |
|-----------------------------|-------------------|--------------------------|--------|
| Injection Name: | 1-Bromopropene ID | Run Time (min): | 20.00 |
| Vial Number: | 7 | Injection Volume: | 1.00 |
| Injection Type: | Unknown | Channel: | GC_1 |
| Comment: | | Wavelength: | n.a. |
| Instrument Method: | Finkelstein 1 | Bandwidth: | n.a. |
| Processing Method: | Quantitative | Dilution Factor: | 1.0000 |
| Injection Date/Time: | 04/May/23 17:12 | Sample Weight: | 1.0000 |

Chromatogram



Integration Results

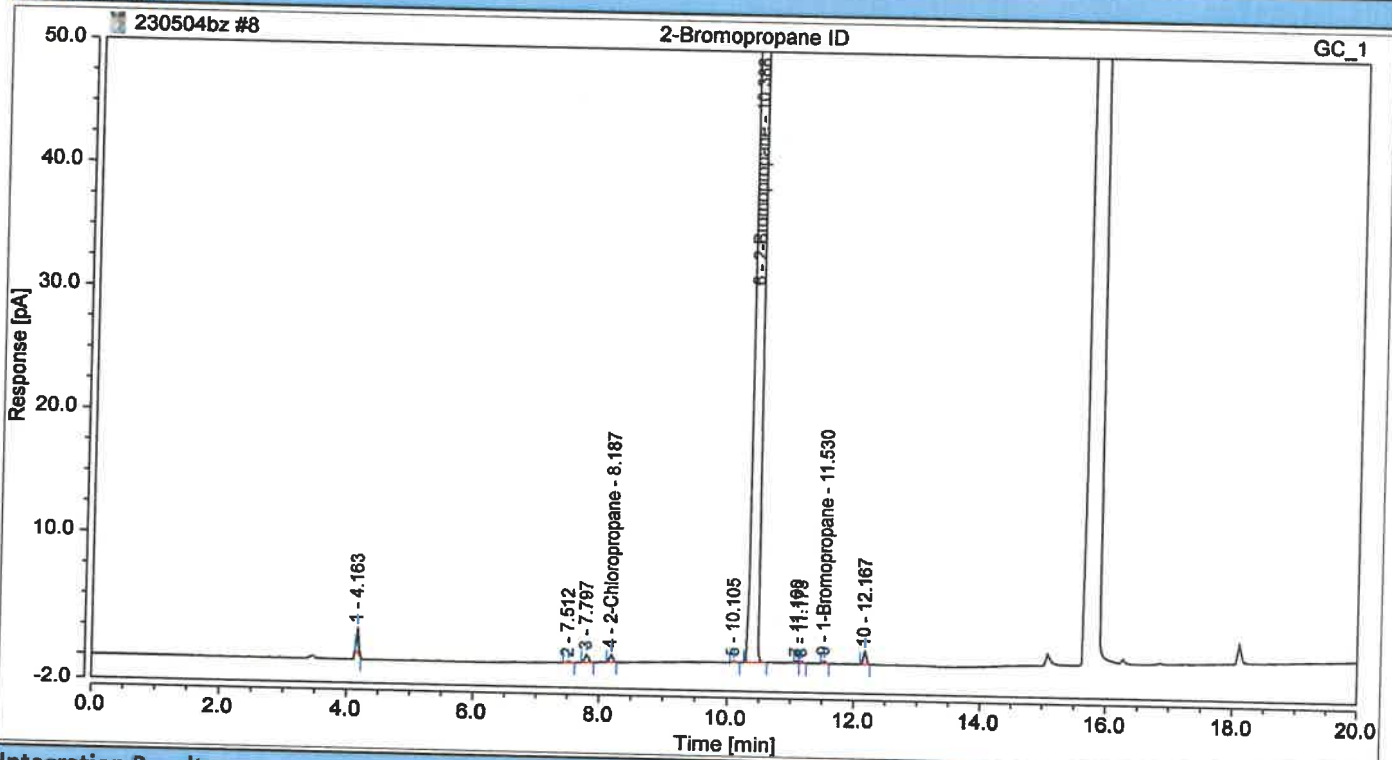
| No. | Peak Name | Retention Time min | Area pA*min | Height pA | Resolution (USP) To Next | Tailing USP | S/N |
|--------|-----------------|-----------------------|----------------|--------------|-----------------------------|-------------|--------|
| 1 | | 4.168 | 0.00683 | 0.156 | n.a. | 1.0 | 18 |
| 2 | | 6.643 | 0.00371 | 0.045 | 17.9 | 1.4 | 4 |
| 3 | 2-Chloropropane | 8.185 | 0.02814 | 0.432 | 13.9 | 1.0 | 31 |
| 4 | 1-Chloropropane | 9.430 | 0.01889 | 0.335 | 1.5 | 0.9 | 23 |
| 5 | | 9.563 | 0.02096 | 0.346 | 9.1 | 1.3 | 24 |
| 6 | 2-Bromopropene | 10.390 | 0.49868 | 8.744 | 8.2 | 1.0 | 527 |
| 7 | | 11.098 | 0.00988 | 0.188 | 1.8 | 1.0 | 19 |
| 8 | | 11.248 | 0.03404 | 0.591 | 3.4 | 1.4 | 52 |
| 9 | 1-Bromopropene | 11.527 | 109.30548 | 2154.439 | 17.5 | 1.0 | 112725 |
| n.a. | 2-Iodopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 10 | | 12.935 | 0.01577 | 0.305 | 3.5 | 1.1 | 37 |
| 11 | 1,4-Dioxane | 13.208 | 0.08259 | 1.716 | 5.1 | 1.1 | 143 |
| 12 | 1-Iodopropane | 13.597 | 0.04288 | 0.870 | n.a. | 1.0 | 72 |
| n.a. | DMAc | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| Total: | | | 110.068 | 2168.168 | 82.04 | 13.20 | |

Chromatogram and Results

Injection Details

| | | | |
|-----------------------------|-------------------|--------------------------|--------|
| Injection Name: | 2-Bromopropane ID | Run Time (min): | 20.00 |
| Vial Number: | 8 | Injection Volume: | 1.00 |
| Injection Type: | Unknown | Channel: | GC_1 |
| Comment: | | Wavelength: | n.a. |
| Instrument Method: | Finkelstein 1 | Bandwidth: | n.a. |
| Processing Method: | Quantitative | Dilution Factor: | 1.0000 |
| Injection Date/Time: | 04/May/23 17:42 | Sample Weight: | 1.0000 |

Chromatogram



Integration Results

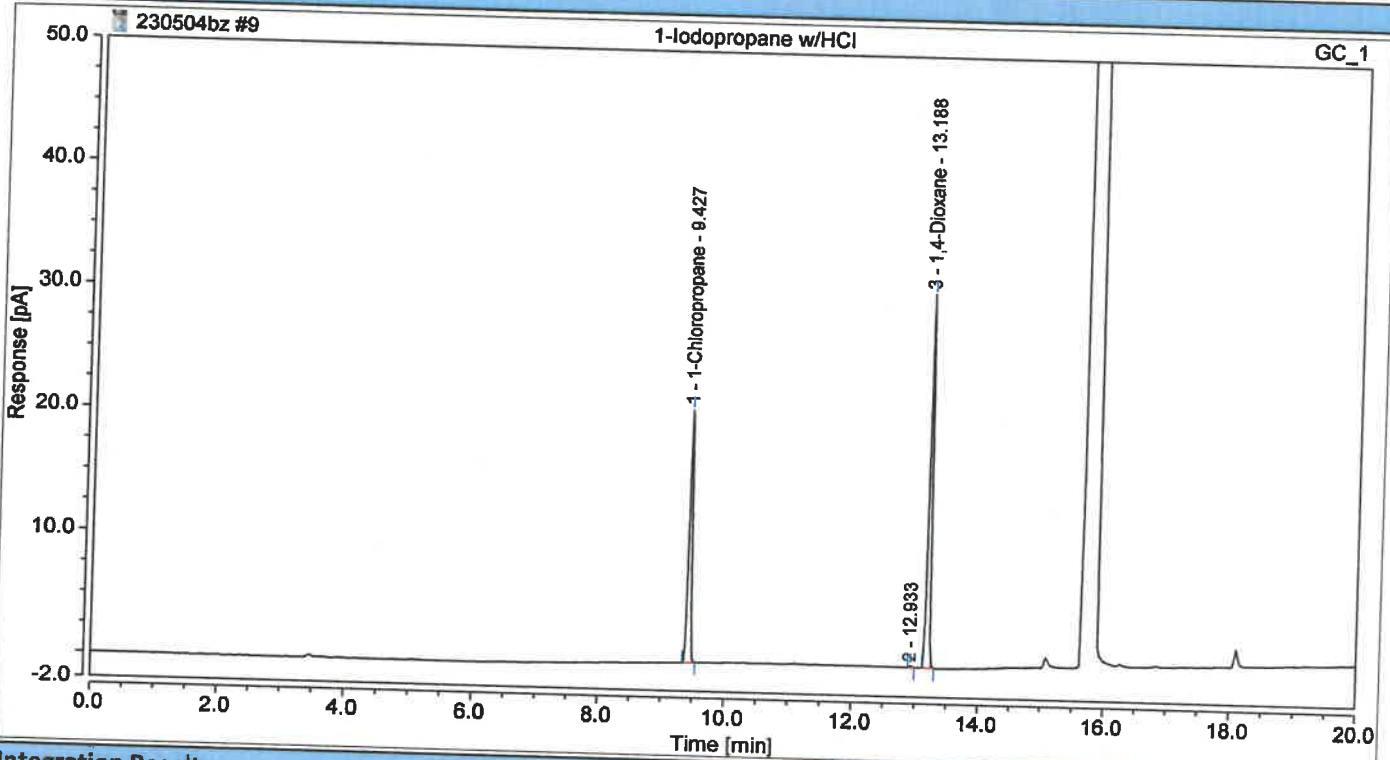
| No. | Peak Name | Retention Time min | Area pA*min | Height pA | Resolution (USP) To Next | Tailing USP | S/N |
|--------|-----------------|-----------------------|----------------|--------------|-----------------------------|-------------|-------|
| 1 | | 4.163 | 0.09411 | 2.163 | 39.0 | 1.1 | 83 |
| 2 | | 7.512 | 0.00622 | 0.087 | 2.8 | 1.3 | 11 |
| 3 | | 7.797 | 0.04388 | 0.684 | 3.9 | 1.1 | 30 |
| 4 | 2-Chloropropane | 8.187 | 0.04045 | 0.648 | 21.2 | 1.0 | 29 |
| n.a. | 1-Chloropropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 5 | | 10.105 | 0.00654 | 0.111 | 3.3 | 0.9 | 10 |
| 6 | 2-Bromopropane | 10.388 | 113.32527 | 1996.522 | 9.5 | 1.0 | 97327 |
| 7 | | 11.100 | 0.00738 | 0.157 | 1.1 | n.a. | 10 |
| 8 | | 11.173 | 0.00655 | 0.127 | 4.2 | n.a. | 8 |
| 9 | 1-Bromopropane | 11.530 | 0.00981 | 0.186 | 7.2 | 1.0 | 15 |
| 10 | | 12.167 | 0.05543 | 1.112 | n.a. | 1.0 | 119 |
| n.a. | 2-Iodopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| n.a. | 1,4-Dioxane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| n.a. | 1-Iodopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| n.a. | DMAC | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| Total: | | | 113.596 | 2001.797 | 92.21 | 8.59 | |

Chromatogram and Results

Injection Details

| | | | |
|----------------------|---------------------|-------------------|--------|
| Injection Name: | 1-Iodopropane w/HCl | Run Time (min): | 20.00 |
| Vial Number: | 9 | Injection Volume: | 1.00 |
| Injection Type: | Unknown | Channel: | GC_1 |
| Comment: | | Wavelength: | n.a. |
| Instrument Method: | Finkelstein 1 | Bandwidth: | n.a. |
| Processing Method: | Quantitative | Dilution Factor: | 1.0000 |
| Injection Date/Time: | 04/May/23 18:12 | Sample Weight: | 1.0000 |

Chromatogram



Integration Results

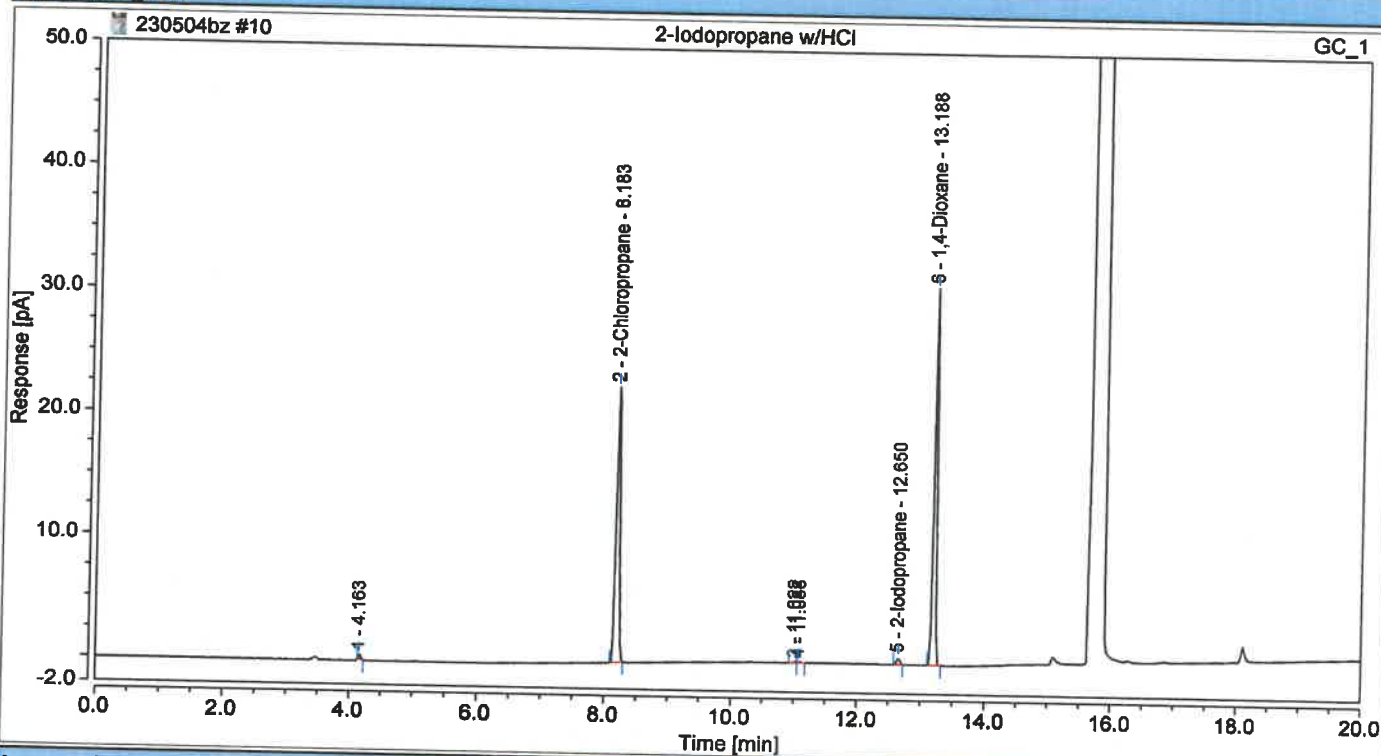
| No. | Peak Name | Retention Time min | Area pA*min | Height pA | Resolution (USP) To Next | Tailing USP | S/N |
|--------|-----------------|-----------------------|----------------|--------------|-----------------------------|-------------|------|
| n.a. | 2-Chloropropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 1 | 1-Chloropropane | 9.427 | 1.13449 | 20.519 | 43.5 | 1.0 | 1269 |
| n.a. | 2-Bromopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| n.a. | 1-Bromopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| n.a. | 2-Iodopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 2 | | 12.933 | 0.00745 | 0.161 | 3.4 | 1.1 | 16 |
| 3 | 1,4-Dioxane | 13.188 | 1.48436 | 30.370 | n.a. | 1.0 | 1676 |
| n.a. | 1-Iodopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| n.a. | DMAc | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| Total: | | | 2.626 | 51.049 | 46.82 | 3.16 | |

Chromatogram and Results

Injection Details

| | | | |
|-----------------------------|---------------------|--------------------------|--------|
| Injection Name: | 2-Iodopropane w/HCl | Run Time (min): | 20.00 |
| Vial Number: | 10 | Injection Volume: | 1.00 |
| Injection Type: | Unknown | Channel: | GC_1 |
| Comment: | | Wavelength: | n.a. |
| Instrument Method: | Finkelstein 1 | Bandwidth: | n.a. |
| Processing Method: | Quantitative | Dilution Factor: | 1.0000 |
| Injection Date/Time: | 04/May/23 18:42 | Sample Weight: | 1.0000 |

Chromatogram



Integration Results

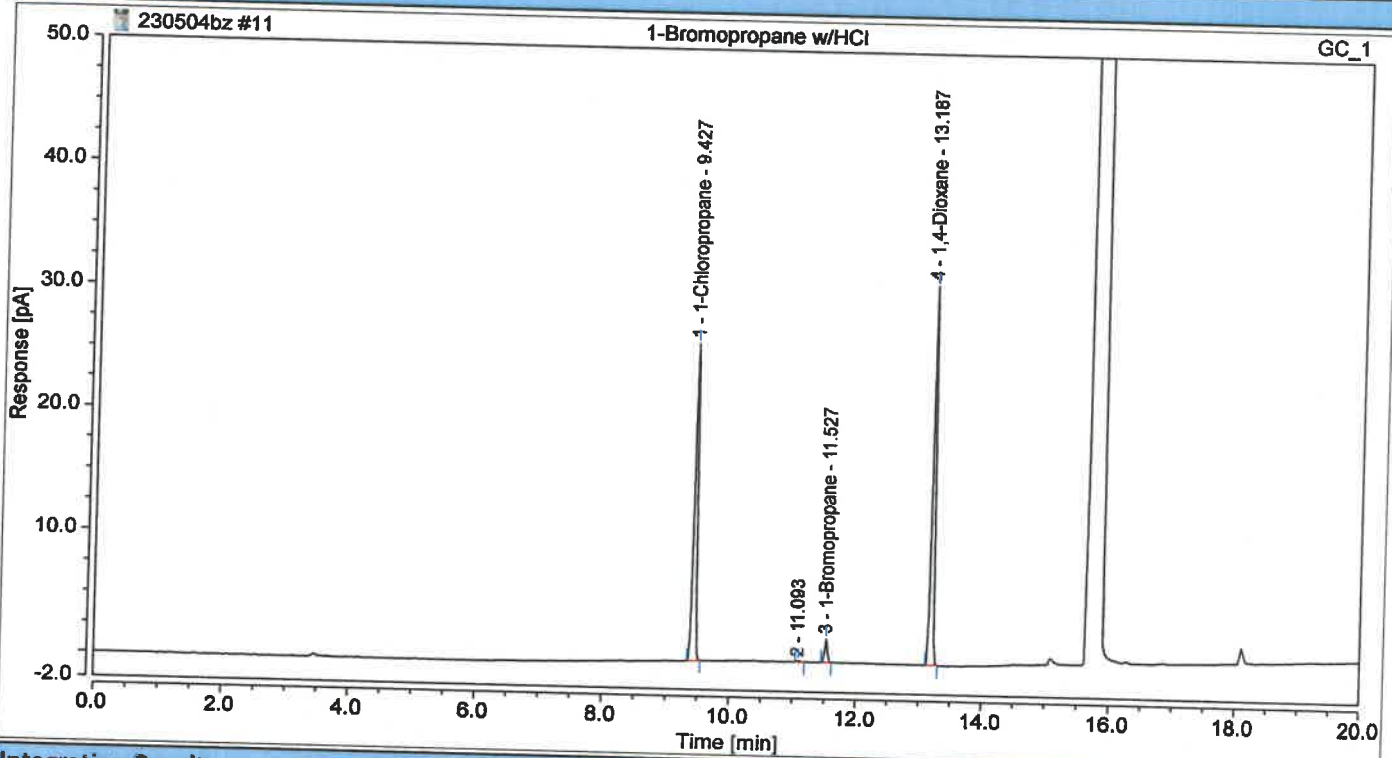
| No. | Peak Name | Retention Time min | Area pA*min | Height pA | Resolution (USP) To Next | Tailing USP | S/N |
|--------|-----------------|-----------------------|----------------|--------------|-----------------------------|-------------|------|
| 1 | | 4.163 | 0.01863 | 0.425 | n.a. | 1.2 | 51 |
| 2 | 2-Chloropropane | 8.183 | 1.41215 | 22.370 | 32.9 | 1.0 | 885 |
| n.a. | 1-Chloropropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| n.a. | 2-Bromopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 3 | | 11.022 | 0.00553 | 0.092 | 0.9 | n.a. | n.a. |
| 4 | | 11.088 | 0.00586 | 0.094 | 19.9 | n.a. | 6 |
| n.a. | 1-Bromopropane | n.a. | n.a. | n.a. | n.a. | n.a. | 7 |
| 5 | 2-Iodopropane | 12.650 | 0.03114 | 0.590 | 6.9 | 1.0 | n.a. |
| 6 | 1,4-Dioxane | 13.188 | 1.48822 | 30.612 | n.a. | 1.0 | 52 |
| n.a. | 1-Iodopropane | n.a. | n.a. | n.a. | n.a. | n.a. | 2136 |
| n.a. | DMAc | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| Total: | | | 2.962 | 54.184 | 60.62 | 4.20 | |

Chromatogram and Results

Injection Details

| | | | |
|----------------------|----------------------|-------------------|--------|
| Injection Name: | 1-Bromopropane w/HCl | Run Time (min): | 20.00 |
| Vial Number: | 11 | Injection Volume: | 1.00 |
| Injection Type: | Unknown | Channel: | GC_1 |
| Comment: | | Wavelength: | n.a. |
| Instrument Method: | Finkelstein 1 | Bandwidth: | n.a. |
| Processing Method: | Quantitative | Dilution Factor: | 1.0000 |
| Injection Date/Time: | 04/May/23 19:12 | Sample Weight: | 1.0000 |

Chromatogram



Integration Results

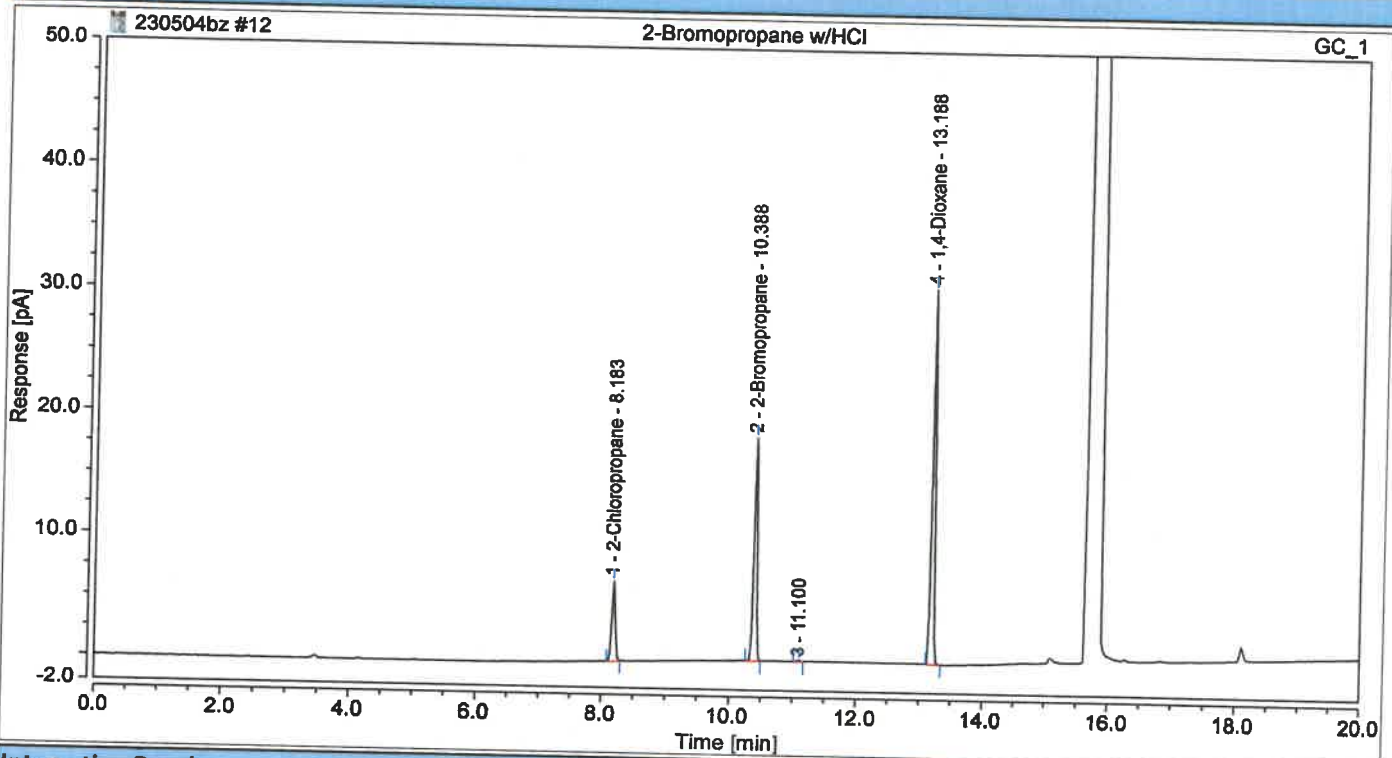
| No. | Peak Name | Retention Time min | Area pA*min | Height pA | Resolution (USP) To Next | Tailing USP | S/N |
|---------------|-----------------|--------------------|--------------|---------------|--------------------------|-------------|------|
| n.a. | 2-Chloropropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 1 | 1-Chloropropane | 9.427 | 1.43375 | 25.783 | 22.0 | 1.0 | 2406 |
| n.a. | 2-Bromopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 2 | | 11.093 | 0.00579 | 0.100 | 6.0 | 1.2 | 8 |
| 3 | 1-Bromopropane | 11.527 | 0.09773 | 1.901 | 20.9 | 1.0 | 193 |
| n.a. | 2-Iodopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 4 | 1,4-Dioxane | 13.187 | 1.49118 | 30.834 | n.a. | 1.0 | 1797 |
| n.a. | 1-Iodopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| n.a. | DMAc | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| Total: | | | 3.028 | 58.618 | 48.89 | 4.24 | |

Chromatogram and Results

Injection Details

| | | | |
|----------------------|----------------------|-------------------|--------|
| Injection Name: | 2-Bromopropane w/HCl | Run Time (min): | 20.00 |
| Vial Number: | 12 | Injection Volume: | 1.00 |
| Injection Type: | Unknown | Channel: | GC_1 |
| Comment: | | Wavelength: | n.a. |
| Instrument Method: | Finkelstein 1 | Bandwidth: | n.a. |
| Processing Method: | Quantitative | Dilution Factor: | 1.0000 |
| Injection Date/Time: | 04/May/23 19:42 | Sample Weight: | 1.0000 |

Chromatogram



Integration Results

| No. | Peak Name | Retention Time min | Area pA*min | Height pA | Resolution (USP) To Next | Tailing USP | S/N |
|---------------|-----------------|-----------------------|----------------|---------------|-----------------------------|-------------|------|
| 1 | 2-Chloropropane | 8.183 | 0.40575 | 6.466 | 23.0 | 1.0 | 244 |
| n.a. | 1-Chloropropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 2 | 2-Bromopropane | 10.388 | 1.03162 | 18.148 | 8.7 | 1.0 | 965 |
| 3 | 1-Bromopropane | 11.100 | 0.00547 | 0.094 | 27.4 | 1.0 | 9 |
| n.a. | 2-Iodopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| 4 | 1,4-Dioxane | 13.188 | 1.49576 | 30.471 | n.a. | 1.0 | 2270 |
| n.a. | 1-Iodopropane | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| n.a. | DMAc | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| Total: | | | 2.939 | 55.180 | 59.07 | 4.04 | |

Processing Method: Quantitative
Last Update Operator: bzielke

Overview

Name Quantitative
Data Vault chrom72
Path chrom://grachromdc01/chrom72/E511/Finkelstein/R&D/230504bz.seq/Quantitative.procmeth
Created 3/1/2023 12:06:16 PM -06:00 bzielke
Last Update 9/21/2023 9:52:14 AM -05:00 bzielke

Detection Settings

Algorithm: **Cobra**
 Baseline Noise Range
 Auto Range **Yes**
 Start Time: **0.000 [min]**
 End Time: **1.000 [min]**

Cobra Smoothing Width: **Auto [min]**
 Consider Void Peak **No**

| Ret. Time | Param. Name | Param. Value | Inj. Type | Channel |
|--------------|----------------------|--------------|-----------|--------------|
| <Initial> | Consider Void Peak | Off | Any | All Channels |
| <Initial> | Baseline Noise Range | Auto | Any | All Channels |
| <Initial> | Smoothing Width | Auto | Any | All Channels |
| 1.214 [min] | Inhibit Integration | On | Any | All Channels |
| 4.129 [min] | Inhibit Integration | Off | Any | All Channels |
| 14.000 [min] | Inhibit Integration | On | Any | All Channels |

Component Table

| Name | Ret. Time | Window | Channel | Eval. Type | Stand. Meth. | Cal. Type | Conc. Unit | Factor | Peak Type |
|-----------------|-----------|----------|--------------|------------|--------------|-----------------|------------|----------|------------|
| 2-Chloropropane | 8.180 | 0.100 AF | All Channels | Area | External | Lin, WithOffset | | 1.000000 | Autodetect |
| 1-Chloropropane | 9.400 | 0.100 AF | All Channels | Area | External | Lin, WithOffset | | 1.000000 | Autodetect |
| 2-Bromopropane | 10.380 | 0.100 AF | All Channels | Area | External | Lin, WithOffset | | 1.000000 | Autodetect |
| 1-Bromopropane | 11.520 | 0.100 AF | All Channels | Area | External | Lin, WithOffset | | 1.000000 | Autodetect |
| 2-Iodopropane | 12.650 | 0.100 AF | All Channels | Area | External | Lin, WithOffset | | 1.000000 | Autodetect |
| 1,4-Dioxane | 13.200 | 0.100 AF | All Channels | Area | External | Lin, WithOffset | | 1.000000 | Autodetect |
| 1-Iodopropane | 13.597 | 0.100 AF | All Channels | Area | External | Lin, WithOffset | | 1.000000 | Autodetect |
| DMAc | 15.700 | 0.100 AF | All Channels | Area | External | Lin, WithOffset | | 1.000000 | Autodetect |

| Group | Low.Conf.Prob | Up.Conf.Prob | Comment | Charge |
|-------|---------------|--------------|---------|--------|
| | 99.5 % | 99.5 % | | |
| | 99.5 % | 99.5 % | | |
| | 99.5 % | 99.5 % | | |
| | 99.5 % | 99.5 % | | |
| | 99.5 % | 99.5 % | | |
| | 99.5 % | 99.5 % | | |
| | 99.5 % | 99.5 % | | |
| | 99.5 % | 99.5 % | | |
| | 99.5 % | 99.5 % | | |

Name Finkelstein 1
Data Vault chrom72
Path chrom://grachromdc01/chrom72/E511/Finkelstein/R&D/230504bz.
seq/Finkelstein 1.instmeth
Comment
Description
Run time 20.000 [min]
Instrument E511_GC on lab-pc23
Created 3/1/2023 12:04:13 PM -06:00 bzielke
Last Update 3/3/2023 11:45:19 AM -06:00 bzielke

| Stage | Time min | Command | Value | Comment |
|---------------------------|-------------|----------------------------------|----------------------------|---------|
| Instrument Setup | | | | |
| | initial | GC.BackInlet.Mode | Split | |
| | | GC.GC_1.MakeupFlow.Nominal | 30.0 [ml/min] | |
| | | GC.Column2.Length | 60.00 [m] | |
| | | GC.Column2.Diameter | 320.00 [µm] | |
| | | GC.Column2.FilmThickness | 3.00 [µm] | |
| | | GC.Column2.Inlet | Back_Inlet | |
| | | GC.Column2.FlowMode | PressureCtrl | |
| | | GC.BackInlet.SplitRatio | 5.0 | |
| | | GC.BackInlet.SeptumPurgeCtrl | Off | |
| | | GC.BackInlet.Temperature.Nominal | 180.0 [°C] | |
| | | GC.BackInlet.TempCtrl | On | |
| | | GC.BackInlet.GasSaver | Off | |
| | | GC.GC_1.Data_Collection_Rate | 10 [Hz] | |
| | | GC.GC_1.LitOffset | 0.5 [pA] | |
| | | GC.GC_1.Flame | On | |
| | | GC.GC_1.Electrometer | On | |
| | | GC.GC_1.H2Flow.Nominal | 40.0 [ml/min] | |
| | | GC.GC_1.H2FlowCtrl | On | |
| | | GC.GC_1.AirFlow.Nominal | 400.0 [ml/min] | |
| | | GC.GC_1.AirFlowCtrl | On | |
| | | GC.GC_1.MakeupMode | Makeup | |
| | | GC.GC_1.MakeupFlowCtrl | On | |
| | | GC.GC_1.Temperature.Nominal | 300.0 [°C] | |
| | | GC.GC_1.TempCtrl | On | |
| | | GC.TempCtrl | On | |
| | | GC.BackInlet.FlowCtrl | On | |
| Equilibration | | | | |
| | 0.000 | GC.Temperature.Nominal | 40.0 [°C] | |
| | | GC.Column2.Pressure.Nominal | 18.0000 [psi] | |
| Inject Preparation | | | | |
| | 0.000 | GC.GC_1.Autozero | | |
| | | Wait | GC.Ready | |
| Inject | | | | |
| | 0.000 | HS_Sampler.Inject | | |
| Start Run | | | | |
| | 0.000 | GC.GC_1.AcqOn | | |
| Run | | | | |
| | 0.000 | | Duration = 20.000 [min] | |

| | | | |
|----------|--------|-----------------------------|---------------|
| | 6.000 | | |
| | | GC.Temperature.Nominal | 40.0 [°C] |
| | 14.400 | | |
| | | GC.Temperature.Nominal | 250.0 [°C] |
| | 20.000 | | |
| | | GC.Temperature.Nominal | 250.0 [°C] |
| Stop Run | | | |
| | 20.000 | | |
| | | GC.GC_1.AcqOff | |
| | | GC.Column2.Pressure.Nominal | 18.0000 [psi] |
| End | | | |