Analytical Repeatability, Accuracy, and Robustness of Instant Connect GC Modules

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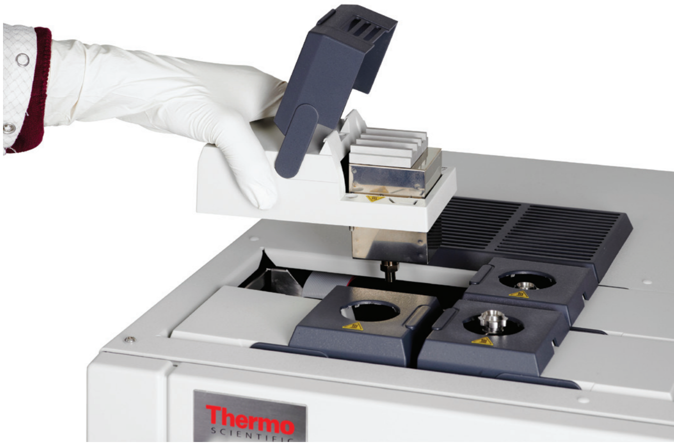
Key Words
TRACE 1300 Series GC, Instant Connect modules, SSL, PTV, FID, ECD, TCD, NPD

Introduction

GC injectors and detectors are considered fundamental components of a gas chromatography system. In modern instrumentation, they consist of the mechanical parts, the inner body with all tubing for gas connections, pneumatics, and electronic controls. Selection of an appropriate injector and detector is based on application requirements. Changing a system configuration to follow a new analytical need or application is a complex operation, requiring specialized service assistance and often resulting in a new system requirement.

The Thermo Scientific™ TRACE™ 1300 Series GC is the first GC instrument on the market that has transcended this design model. Similar to the long-established modularity in HPLC, it makes the fundamental instrument components (the injector and detector) available as independent sub-systems, which are combined to produce the desired analytical layout. The level of modularity of the TRACE 1300 Series GC allows users to rapidly adapt the instrument configuration to new application and/or workload requirements without consulting a service engineer.

This new GC modularity is implemented in the TRACE 1300 Series GC in the form of a full range of injector and detector modules, which are easy and quick to install and swap. These modules, termed *Instant Connect*, incorporate all relevant pneumatic hardware and electronic parts necessary for making the injector or the detector a fully self-sufficient sub-unit of the instrument. All electronic circuits and pneumatic controls are integrated into the injector body or detector cell, and enclosed into a light, 17 cm x 10 cm x 6 cm, easy-to-handle housing. Each module stores all specific electronic and pneumatic calibration information, minimizing module-to-module performance variation. The modules are plugged into the top part of the GC, are automatically configured into the system, and connected to the gas supply lines. Installing a module takes only two minutes: the time needed to fix three retaining screws and slide the new injector or detector module in place.



Laboratories can benefit from the versatility provided by this "Instant Connect" modularity in several ways:

- Expanding instrument capability at any time, by adding a new injector or detector module to run a new method
- Upgrading a GC from single to multiple channels to satisfy rapid incremental business needs and enhance laboratory productivity
- Replacing contaminated injectors or detectors quickly with clean ones and running samples in a few minutes, while conducting full maintenance and cleaning when the laboratory schedule allows
- Sharing injectors and detectors with different TRACE 1300 Series GC units in a lab depending on the application

The list of "Instant Connect" modules includes Split/Splitless (SSL) and Programmable Temperature Vaporizing (PTV) injectors both in the standard and backflush configuration and all standard GC detectors: Flame Ionization Detector (FID), Electron Capture Detector (ECD), Thermal Conductivity Detector (TCD), and Nitrogen Phosphorous Detector (NPD).

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This technical note describes results obtained from an endurance mechanical test performed on "Instant Connect" modules and typical analytical reproducibility and accuracy provided by the TRACE 1300 Series GC. Analytical reproducibility was also measured after switching modules of the same type in the same application, as might happen when an injector module is replaced with a new one in a routine laboratory to keep a contaminated instrument up and running.

Experimental

A TRACE 1310 GC instrument equipped with various SSL and FID modules was used in all of the experiments, and all "Instant Connect" modules are identified by specific serial numbers for easier tracking in the lab. Four different modules were alternated in these experiments.

The GC was equipped with a Thermo Scientific AS 1310 liquid autosampler. All tests were performed using a synthetic mix of normal alkanes ranging from C_{10} to C_{40} in hexane, at a concentration of approximately 10 ppm (10 ng/µL) and using helium as carrier gas. A 1 µL aliquot of sample was injected in splitless mode into a standard glass-wool packed tapered liner, while the injector temperature was maintained at 300 °C. Splitless time was 0.8 minutes. The FID detector temperature was set to 350 °C.

A Thermo Scientific TraceGOLD™ column TR-5, 15 m x 0.25 mm id x 0.25 µm, was used in all experiments. Oven temperature was set at 50 °C for 0.5 min and then ramped up to 340 °C at 20 °C/min, with two minutes of isothermal time at the final temperature. A Thermo Scientific Dionex™ Chromeleon™ Chromatography Data System was used for setting all method parameters, data acquisition, and data processing.

Results and Discussion

System analytical reproducibility and accuracy

The analytical reproducibility was evaluated using two new "Instant Connect" modules, a SSL injector (module serial number S/N: 712100036) and a FID detector (S/N: 712300088), by injecting the synthetic hydrocarbon mix automatically (ten repetitions). Results in terms of peak area and retention time repeatability are summarized in Tables 1 and 2. No discrimination for both volatile and high boiling compounds was seen. As shown in Table 1 the recovery, calculated using C_{20} peak area as reference, is close to 100% along the full range of volatility. Absolute peak area relative standard deviation is far below 1% for all hydrocarbons. All injector and detector modules incorporate a new generation of miniaturized gas controls. These integrated electronic devices ensure precise control of the inlet pressure and the flow throughout the column, further contributing to the excellent reproducibility of retention times. As indicated in Table 2, the standard deviation is below a thousandth of a minute. This level of reproducibility is a clear indication of the accurate temperature profile and column flow maintained during the ramp and the precise thermo-regulation of the GC oven. Overall results show full recovery of hydrocarbons and excellent data precision.

Module-to-module reproducibility

To simulate a situation where a laboratory needs to quickly replace a module, such as to avoid interrupting instrument throughput for maintenance, the "Instant Connect" SSL injector module (S/N: 712100036) was replaced by a new module (S/N: 712300021). This required cooling and powering down the instrument, disconnecting the column from the original SSL injector module, removing the module and plugging in the new one, connecting the column, and powering up the TRACE 1310 GC again. Electronic gas control permits an automated leak check to be performed to guarantee that no artifacts are introduced by this manual operation. The reduced thermal mass GC design allows a quick recovery of injection-ready conditions after instrument power-up. As a result, the GC was ready to resume analytical injections again in only nine minutes after it was originally powered down. A blank GC cycle was programmed before injecting samples again, which is good practice to ensure the entire flow path was not affected by air introduced during module replacement.

An automated sequence of 10 injections was performed immediately after the module replacement along with collecting data. The instrument was then stopped again, and the FID detector module (S/N: 712300088) was replaced by a new one (S/N: 712300126). After a blank run, another sequence of 10 injections completed the experiments. Tables 3 and 4 and Figure 1 summarize the repeatability results for the three different instrument configurations. Variations in peak area measured as a delta of the average counts are in the range of a few percentages when changing either the injector or the FID detector. Such a variation, for many applications, is well below the required limit of a system suitability check, eliminating the need to recalibrate the GC system as a whole. The retention time variations are in the range of a few hundredths of a minute or even less with no impact on component retention time.

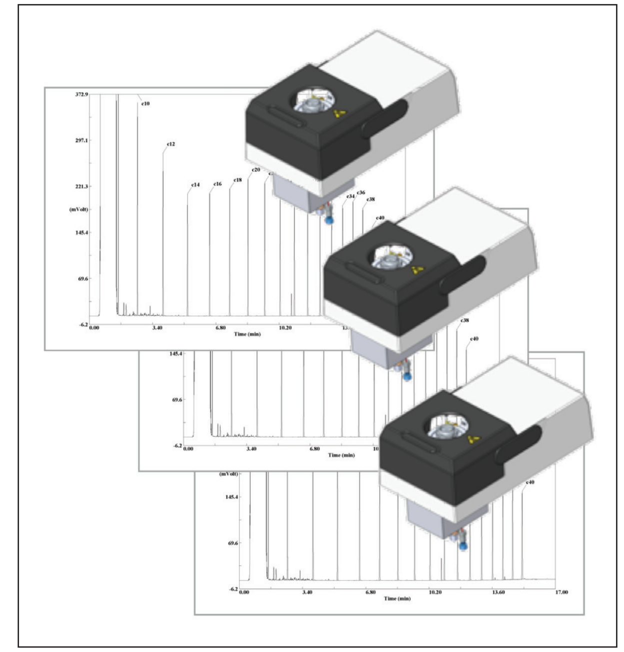

Figure 1. Module-to-module repeatability. Modules store all of their calibration information allowing minimum variation if replaced on a system.

Table 1. Instrument configuration SSL S/N 712100036 and FID S/N 712300088. Absolute peak area RSD% far lower than 1%. Recovery, measured as ratio vs C_{20} average area, at 100% for the whole range of hydrocarbons

SSL 712100036 / FID 712300088	nC10	nC12	nC14	nC16	nC18	nC20	nC22	nC24	nC26	nC28	nC30	nC32	nC34	nC36	nC38	nC40
Injection 1	2600304	2647767	2600941	2619188	2552750	2565809	2543886	2535687	2512699	2527008	2602759	2597807	2534441	2564855	2470270	2525384
Injection 2	2610605	2657856	2601653	2623404	2568557	2579380	2565938	2565679	2545232	2560614	2636265	2629734	2557462	2596729	2495209	2563483
Injection 3	2602666	2653832	2599714	2626029	2553641	2577265	2561577	2558672	2542703	2555406	2632496	2630095	2555743	2580475	2480864	2538819
Injection 4	2636572	2683702	2632897	2656448	2593709	2602685	2584957	2575384	2558834	2574920	2649582	2640082	2566623	2593858	2490053	2549873
Injection 5	2623737	2668679	2617130	2639475	2575209	2588255	2568857	2566489	2551218	2570336	2641759	2632243	2559472	2591033	2487269	2545848
Injection 6	2628675	2671731	2625320	2647746	2586155	2602674	2584119	2578956	2563433	2577945	2652932	2644762	2572449	2600568	2495549	2560371
Injection 7	2633245	2675436	2621623	2640507	2579749	2601553	2603546	2589030	2566470	2580193	2651340	2644782	2575086	2615870	2515378	2552861
Injection 8	2622426	2667773	2618401	2631007	2571368	2588047	2571982	2568771	2543992	2565899	2635937	2628421	2556233	2599156	2491820	2552234
Injection 9	2627383	2675413	2624978	2646945	2578061	2590137	2578171	2582555	2553973	2565982	2637795	2636002	2561603	2598494	2504800	2575965
Injection 10	2621650	2664829	2611668	2634863	2576839	2592681	2577082	2571091	2552087	2567396	2634338	2631176	2560023	2590260	2497076	2558360
Average (counts)	2620726	2666702	2615432	2636561	2573604	2588848	2574011	2569231	2549064	2564570	2637520	2631510	2559913	2593130	2492829	2552320
SD	12355	10941	11571	11889	12894	12092	15913	14767	15078	15223	14298	13319	11161	13430	12358	13908
RSD %	0.47%	0.41%	0.44%	0.45%	0.50%	0.47%	0.62%	0.57%	0.59%	0.59%	0.54%	0.51%	0.44%	0.52%	0.50%	0.54%
Recovery %	101%	103%	101%	102%	99%	100%	99%	99%	98%	99%	102%	102%	99%	100%	96%	99%

Table 2. Retention time standard deviation in the range of 1/1000 minute

	nC10	nC12	nC14	nC16	nC18	nC20	nC22	nC24	nC26	nC28	nC30	nC32	nC34	nC36	nC38	nC40
Injection 1	2.562	3.935	5.253	6.445	7.525	8.506	9.408	10.237	11.005	11.717	12.385	13.010	13.598	14.153	14.680	15.190
Injection 2	2.562	3.933	5.252	6.445	7.525	8.508	9.408	10.237	11.005	11.718	12.385	13.010	13.598	14.153	14.678	15.188
Injection 3	2.562	3.935	5.252	6.445	7.523	8.505	9.407	10.233	11.003	11.715	12.383	13.007	13.597	14.152	14.680	15.189
Injection 4	2.562	3.935	5.253	6.445	7.525	8.508	9.408	10.237	11.005	11.718	12.385	13.008	13.598	14.152	14.678	15.188
Injection 5	2.562	3.933	5.252	6.445	7.525	8.508	9.408	10.237	11.003	11.717	12.385	13.010	13.600	14.154	14.678	15.191
Injection 6	2.562	3.933	5.252	6.445	7.525	8.508	9.408	10.237	11.005	11.717	12.385	13.010	13.598	14.153	14.680	15.189
Injection 7	2.562	3.933	5.252	6.447	7.525	8.507	9.407	10.237	11.003	11.718	12.385	13.008	13.597	14.153	14.681	15.190
Injection 8	2.562	3.933	5.252	6.445	7.525	8.507	9.408	10.235	11.003	11.717	12.385	13.008	13.598	14.150	14.680	15.190
Injection 9	2.560	3.932	5.250	6.443	7.523	8.506	9.408	10.235	11.002	11.717	12.385	13.008	13.597	14.153	14.682	15.191
Injection 10	2.562	3.933	5.252	6.445	7.525	8.508	9.408	10.237	11.005	11.718	12.385	13.010	13.597	14.152	14.682	15.188
Average (minutes)	2.562	3.934	5.252	6.445	7.525	8.507	9.408	10.236	11.004	11.717	12.385	13.009	13.598	14.153	14.680	15.190
SD	0.0005	0.0009	0.0009	0.0008	0.0007	0.0012	0.0007	0.0012	0.0012	0.0011	0.0005	0.0012	0.0011	0.0012	0.0013	0.0010
RSD %	0.02%	0.02%	0.02%	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%	0.00%	0.01%	0.01%	0.01%	0.01%	0.01%

Table 3. Variation in peak area as effect of module swap. All variations are in the range of few % changing either the inlet or the FID detector

Original instrument configuration SSL s/n 712100036 and FID s/n 712300088																
	nC10	nC12	nC14	nC16	nC18	nC20	nC22	nC24	nC26	nC28	nC30	nC32	nC34	nC36	nC38	nC40
Average (counts)	2620726	2666702	2615432	2636561	2573604	2588848	2574011	2569231	2549064	2564570	2637520	2631510	2559913	2593130	2492829	2552320
SD	12355	10941	11571	11889	12894	12092	15913	14767	15078	15223	14298	13319	11161	13430	12358	13908
RSD %	0.47%	0.41%	0.44%	0.45%	0.50%	0.47%	0.62%	0.57%	0.59%	0.59%	0.54%	0.51%	0.44%	0.52%	0.50%	0.54%
Change of SSL module - Instrument configuration SSL 712300021 / FID 712300088																
Average (counts)	2705439	2722254	2654680	2680682	2615418	2647035	2626550	2624551	2604909	2618663	2699958	2707570	2658013	2713142	2598635	2604178
SD	8276	7559	8759	9119	11059	11146	12635	14822	13711	16916	16529	17096	12977	10030	12448	10215
RSD %	0.31%	0.28%	0.33%	0.34%	0.42%	0.42%	0.48%	0.56%	0.53%	0.65%	0.61%	0.63%	0.49%	0.37%	0.48%	0.39%
Variation %	-3.2%	-2.1%	-1.5%	-1.7%	-1.6%	-2.2%	-2.0%	-2.2%	-2.2%	-2.1%	-2.4%	-2.9%	-3.8%	-4.6%	-4.2%	-2.0%
Change of FID module - Instrument configuration SSL 712300021 / FID 712300126																
Average (counts)	2752208	2777431	2705697	2728377	2668020	2699389	2678126	2670723	2649792	2665081	2745907	2757795	2703327	2763143	2653118	2666225
SD	13455	15147	15120	11600	15162	14201	15885	15954	14781	15601	11514	14864	10635	13223	15755	11218
RSD %	0.49%	0.55%	0.56%	0.43%	0.57%	0.53%	0.59%	0.60%	0.56%	0.59%	0.42%	0.54%	0.39%	0.48%	0.59%	0.42%
Variation %	-1.7%	-2.0%	-1.9%	-1.8%	-2.0%	-2.0%	-2.0%	-1.8%	-1.7%	-1.8%	-1.7%	-1.9%	-1.7%	-1.8%	-2.1%	-2.4%

Table 4. Variation in retention time as effect of module swap. All variations are in the range of 1/100 of a minute or less, changing either the inlet or the FID detector

Original instrument configuration SSL s/n 712100036 and FID s/n 712300088																
	nC10	nC12	nC14	nC16	nC18	nC20	nC22	nC24	nC26	nC28	nC30	nC32	nC34	nC36	nC38	nC40
Average (minutes)	2.562	3.934	5.252	6.445	7.525	8.507	9.408	10.236	11.004	11.717	12.385	13.009	13.598	14.153	14.680	15.190
SD	0.0005	0.0009	0.0009	0.0008	0.0007	0.0012	0.0007	0.0012	0.0012	0.0011	0.0005	0.0012	0.0011	0.0012	0.0013	0.0010
RSD %	0.02%	0.02%	0.02%	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%	0.00%	0.01%	0.01%	0.01%	0.01%	0.01%
Change of SSL module - Instrument configuration SSL 712300021 / FID 712300088																
Average (minutes)	2.566	3.938	5.255	6.448	7.527	8.509	9.410	10.238	11.005	11.719	12.386	13.011	13.599	14.154	14.679	15.188
SD	0.0006	0.0012	0.0007	0.0004	0.0009	0.0007	0.0007	0.0014	0.0007	0.0015	0.0009	0.0015	0.0009	0.0014	0.0015	0.0014
RSD %	0.02%	0.03%	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%
Variation %	-0.2%	-0.1%	-0.1%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
Change of FID module - Instrument configuration SSL 712300021 / FID 712300126																
Average (minutes)	2.563	3.935	5.254	6.446	7.525	8.508	9.408	10.237	11.004	11.718	12.384	13.011	13.598	14.154	14.679	15.186
SD	0.0007	0.0007	0.0010	0.0007	0.0014	0.0008	0.0009	0.0014	0.0010	0.0009	0.0009	0.0010	0.0018	0.0007	0.0014	0.0019
RSD %	0.03%	0.02%	0.02%	0.01%	0.02%	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%
Variation %	0.1%	0.1%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%

Injector module endurance test

Module robustness was tested mechanically by having ten different operators repeatedly insert and remove the module. The operators had widely varied skills and knowledge, with some subjects having no prior GC experience. Two operators were from the shipping department, and two worked in order processing. Two had limited GC knowledge and were the Quality Manager and Product Manager of a different product line. Finally, two engineers and two GC scientists that also presided over all of the tests participated. The module subjected to the test was the “Instant Connect” SSL injector (S/N: 712300021), and the sequence applied by each operator included powering off the GC, removing the module, inserting the module, and powering up the GC until it reached stand-by condition. The total average test sequence time was six minutes. Each operator repeated this cycle ten times.

After each operator finished his or her cycle, the column was connected again to the SSL injector and FID detector, followed by a double blank run. Ten automated injections of the hydrocarbon mix completed the test. Tables 5 and 6 include the results of the last two runs performed before starting the ruggedness test and the two initial runs of the new sequence. It is useful to note that the new sequence was started the day after the last sequence of injections was recorded. The variations of both absolute peak areas and retention times indicate the module performed perfectly without requiring any maintenance.

Table 5. Variation in peak area before and after 100 times module replacement cycle

	nC10	nC12	nC14	nC16	nC18	nC20	nC22	nC24	nC26	nC28	nC30	nC32	nC34	nC36	nC38	nC40
Injection 9 before IC swap	2754987	2779540	2709468	2720590	2662466	2694642	2671418	2666034	2640542	2660383	2748956	2756412	2705301	2768808	2658421	2670870
Injection 10 before IC swap	2751265	2775027	2708032	2732281	2677453	2705799	2688053	2684329	2667261	2684684	2755387	2771243	2709754	2772642	2651536	2665536
Injection 1 after IC swap	2767372	2791927	2719553	2738439	2664499	2693367	2672357	2657758	2643338	2655810	2738028	2745997	2704789	2768416	2664390	2670998
Injection 2 after IC swap	2756768	2787601	2711585	2738364	2687682	2720242	2699762	2690563	2663741	2677520	2756966	2774421	2711745	2765971	2664631	2676359
Variation	-0.59%	-0.61%	-0.43%	-0.23%	0.48%	0.46%	0.58%	0.99%	0.90%	1.08%	0.63%	0.91%	0.18%	0.15%	-0.48%	-0.20%

Table 6. Variation in retention time before and after 100 times module replacement cycle

	nC10	nC12	nC14	nC16	nC18	nC20	nC22	nC24	nC26	nC28	nC30	nC32	nC34	nC36	nC38	nC40
Injection 9 before IC swap	2.562	3.935	5.253	6.447	7.525	8.507	9.408	10.235	11.003	11.717	12.383	13.010	13.598	14.153	14.678	15.185
Injection 10 before IC swap	2.563	3.933	5.255	6.445	7.523	8.508	9.407	10.237	11.005	11.717	12.383	13.012	13.595	14.154	14.677	15.185
Injection 1 after IC swap	2.563	3.935	5.253	6.447	7.525	8.507	9.408	10.238	11.003	11.718	12.385	13.010	13.598	14.153	14.678	15.183
Injection 2 after IC swap	2.563	3.935	5.253	6.447	7.523	8.508	9.407	10.237	11.003	11.718	12.385	13.012	13.600	14.155	14.680	15.187
Variation	-0.03%	-0.04%	0.04%	-0.03%	-0.02%	0.01%	-0.02%	-0.02%	0.02%	-0.01%	-0.01%	0.02%	-0.02%	0.00%	-0.01%	0.01%

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Conclusion

The "Instant Connect" modules on the TRACE 1300 Series GC offer important advantages over conventional GC instrumentation, such as maintaining instrument uptime and continuing to run even when an injector or detector must be replaced for maintenance purposes. Additional advantages include the ability to upgrade from a single-channel GC to a double-channel GC to increase instrument productivity and the option to add a new detector module to respond to new application requirements.

The design of "Instant Connect" modules as self-independent components of the GC, which incorporate all mechanical and electronic components with calibration information, permits the user to rapidly remove and install new modules without any service assistance. The modularity of the design provides configuration flexibility never before available in a GC and also maintains the highest reproducibility and ruggedness standards. The test results show that module internal calibration allows module-to-module reproducibility to be within 5% of the variances in absolute peak area and retention times. The compact size and robustness of the module design enable the modules to be repeatedly replaced without impacting instrument performance.